

भारतीय मानक

हाईड्रोमेट्री — धारा एवं नहरों में तलछट — सान्द्रण, कण
साईंज वितरण एवं सापेक्ष घनत्व का निर्धारण
(पहला पुनरीक्षण)

Indian Standard

HYDROMETRY — SEDIMENT IN STREAMS AND
CANALS — DETERMINATION OF CONCENTRATION,
PARTICLE SIZE DISTRIBUTION AND
RELATIVE DENSITY

(*First Revision*)

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FOREWORD

This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Hydrometry Sectional Committee had been approved by the Water Resources Division Council.

Silt, sand, gravel and other insoluble materials transported by the streams and canals as suspended load, bed load and slope wash load pose problems of vital importance with regard to successful operation of many projects concerning flood control, soil conservation, irrigation, navigation and hydropower development, etc. Therefore, to have adequate knowledge of the sediment load characteristics, a systematic determination of concentration, particle size distribution and relative density of the sedimentary materials is highly desirable.

For the analysis of concentration, particle size distribution and sediment transport. This standard was first published in 1971. This standard was based on ISO 4365. Since the publication of this standard development has taken place and advancement in the related technology has been made in the international scenario. In order to ensure compatibility with the revised ISO 4365 : 2005 ‘Liquid flow in open channels — Sediment in streams and canals — Determination of concentration, particle size distribution and relative density’ this revision has been taken up.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 ‘Rules for rounding off numerical values (*revised*)’. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

HYDROMETRY — SEDIMENT IN STREAMS AND CANALS — DETERMINATION OF CONCENTRATION, PARTICLE SIZE DISTRIBUTION AND RELATIVE DENSITY

(*First Revision*)

1 SCOPE

This standard specifies methods for the determination of the concentration, particle size distribution and relative density of sediment in streams and canals.

2 REFERENCES

The following standards contain provisions which through reference in this text constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

<i>IS No.</i>	<i>Title</i>
1191 : 2003	Hydrometric determination — Vocabulary and symbols (<i>second revision</i>)
4890 : 1968	Methods for measurement of suspended sediment in open channels
15360 : 2003	Measurement of liquid flow in open channels — Bed material sampling

3 DEFINITIONS

For the purpose of this standard, the definitions given in IS 1191 and the following shall apply.

3.1 Bed Load — The sediment in almost continuous contact with the bed, carried forward by rolling, sliding or hopping.

3.2 Bed Material — Sediment of which the stream bed is composed.

3.3 Bed Material Load — The part of the total sediment which consists of the bed material whose rate of movement is governed by the transporting capacity of the channel.

3.4 Nominal Diameter — The diameter of a sphere of the same volume as the given particle.

3.5 Projected Diameter — The diameter of the smallest circle that encloses the projected image of a

particle when viewed in the plane of maximum stability.

3.6 Sediment Concentration — The ratio of the mass or volume of dry sediment in a water-sediment mixture to the total mass or volume of the suspension.

NOTE — It is necessary to mention while reporting whether mass concentration or volume concentration is reported.

3.7 Sedimentation Diameter — The diameter of a sphere having the same relative density and terminal settling velocity as a given particle in the same sedimentation fluid.

3.8 Sieve Diameter — The width of a square opening through which the given particles will just pass.

3.9 Relative Density — The ratio of the mass of a given volume of sediment to the mass of an equal volume of water at 4°C.

3.10 Suspended Load — That part of the total sediment transported which is maintained in suspension by turbulence in the flowing water for considerable periods of time without contact with the streambed.

NOTE — The sediment moves practically with the same velocity as that of the flowing water. It is generally expressed as a mass or volume per unit of time.

4 UNITS OF MEASUREMENT

The units of measurement used in this standard are those of the international system of units (SI) and litre.

5 PROPERTIES OF SEDIMENT

5.1 General

The transport of sediment depends as much upon the properties of the sediment as upon the hydraulic characteristics of the flow. The properties of sediment are defined by individual particle characteristics and bulk characteristics.

5.2 Properties of Individual Particles

Sediment size is the most commonly used parameter to designate the properties of individual particles.

While the size of sediment and its packing directly affect the roughness of the bed, the settling velocity of the particles characterises their reaction to flow and governs the movement of the sediment. This in turn depends upon the relative density, shape, the size of the particle and its zeta potential.

Since particles of natural sediment are of irregular shape, a single length or diameter has to be chosen to characterise the size. Four such diameters, that is nominal diameter, projected diameter, sedimentation diameter and sieve diameter, are used for different particle sizes or purposes (for example, sieve diameter for coarse and medium particles, sedimentation diameter for fine particles which are not usually separated by sieves). The nominal diameter has little significance in sediment transport, but is useful in the study of sedimentary deposits.

5.3 Bulk Characteristics

As sediments consist of large numbers of particles differing in size, shape, relative density, settling velocity, etc, it is essential to find some parameters that can represent the characteristics of the group of particles as a whole. Therefore, a sample of sediment is usually divided into classes according to characteristics (size, settling velocity, etc) and the percentage by mass of the total in each class is determined for the particular characteristic. Frequency distribution curves may be drawn from this data and their parameters (mean, standard deviation, etc.) determined.

6 SAMPLING

Samples of suspended sediment shall be collected as specified in IS 4890.

7 SUSPENDED SEDIMENT CONCENTRATION

7.1 Methods for Determination of Suspended Sediment Concentration

7.1.1 General

Suspended sediment concentrations may be determined by any of the following methods:

- Evaporation method,
- Filtration method, and
- Hydrometer method (also used for determination of particle size).

NOTE — Although the evaporation method requires less time, the filtration method has the advantage that the fractions collected can be photographed on the filters and are available for further examination. However, the filtration method is prone to greater loss of material, whereas in the evaporation method the ratio of sample mass to tare mass is small. Therefore, no hard and fast guidelines can be provided for their choice, and each case should be judged on its merits.

7.1.2 Evaporation Method

The evaporation method is specified in Annex A.

7.1.3 Filtration Method

7.1.3.1 The filtration may be carried out using either filter papers in conical glass funnels or glass fibre filter disc in Coors or Gooch crucible, or fritted glass or alundum crucibles, with the application of a vacuum aspirator system to accelerate the passage of the filtrate.

The filtration method using filter papers and funnels is specified in Annex B.

7.1.3.2 Filter discs, glass fibre made without organic bindings, Whatman grade 934 AH or Gelman type A/E or Millipore type AP 40 or equivalent can be used in a suitable filtration apparatus such as 25 ml to 40 ml capacity Coors or Gooch crucible with adapter.

The filtration method using glass-fibre filter disk is specified in Annex C.

7.1.3.3 A fritted glass crucible is made of pyrex or other resistance glass, the base of the crucible is fusion fitted with a porous fritted disc insert. It is available in different porosity grades such as coarse, medium and fine (of pore size 40-60 μm , 10-15 μm and 4-5.5 μm respectively). The particular grade should be selected according to the nature of the sample. The method using fritted glass crucible is specified in Annex C.

7.1.3.4 Filtering alundum crucibles are similar in shape and size to Gooch or fritted glass crucibles, but made of fused aluminium oxide. They are made in three degrees of porosity (coarse, medium and fine). These are plain and ignition types. The particular type and porosity is selected according to the nature of the sample and purpose of use.

The method of using Alundum crucibles is similar to that using glass-fibre filter discs or fritted glass crucibles (see Annex C). However, alundum crucibles are used without the addition of a filtering medium.

The main advantages of using alundum crucibles are:

- Crucibles are light in mass, which facilitates greater sensitivity in weighing operations; and
- Tare masses are less subject to change.

NOTES

1 For samples containing significant quantity of very fine particles, the last two *viz.* fritted glass and alundum crucible methods are less accurate because of loss of some of these particles during filtration and washing stages.

2 In addition to the above mentioned methods, Hydrometer or Gooch crucible with asbestos layer as filtering medium are also used to determine sediment concentration. Hydrometer method, although a rapid method, is not accurate when sediment concentration is not high or they settle rapidly. Moreover the

hydrometer is usually calibrated for 19.4 °C and hence needs to be recalibrated at different temperatures. Regarding use of asbestos layer as filtering medium in Gooch crucibles, many countries no longer allow use of asbestos. Moreover the pore size of asbestos is undefined, depending on the thickness and uniformity of the layer. Some portion of asbestos may get washed out from the filtration layer causing loss in accuracy. The hydrometer method is specified in **D.1.1**.

7.2 Expression of Concentration

The concentration of suspended sediment shall be expressed as the mass or volume of dry sediment per unit mass or volume of suspension (that is mass/volume or mass/mass, etc).

8 PARTICLE SIZE ANALYSIS

8.1 Particle Size Analysis of Suspended Sediment

For the analysis of particle size, suspended sediment may be classified in terms of sedimentation diameter as follows:

- Coarse sediment, comprising particles of diameter greater than 0.25 mm;
- Medium sediment, comprising particles of diameter between 0.062 mm to 0.25 mm; and
- Fine sediment, comprising particles of diameter less than 0.062 mm.

The methods of analysing suspended sediment of these classes are specified in Annex D.

NOTE — In the case of suspended sediment, grading by particle size into divisions finer than 0.062 mm is usually not carried out because of the unimportance of accurately separating the small amount of solid particles that generally exist in suspension. If, however, closer separation between the coarse and medium sediments is required, it may be carried out by the procedure specified in **8.2** for bed load and bed material. Particle size analysis procedure of fine sediment is given in **E.3.5**.

8.2 Particle Size Analysis of Bed Load and Bed Material

For analysis of samples of the bed load or bed material for particle size distribution and mean diameter, samples are classified broadly into those of diameter greater than, and those of diameter less than 0.5 mm. Classification of material in these two ranges is suitable in the computation of bed load.

The methods of analysing bed load and bed material are specified in Annex E.

The particle size distribution of sediment may be determined by sieving (when particles are all coarse), by a combination of sieving and settling velocity, or indirectly by measuring particle-settling velocities in a column of liquid. It would be advantageous to use only one measure of diameter over the entire range of sizes for all sediments, preferably the sedimentation diameter,

but this is not practicable since large particles will settle very rapidly in the sedimentation liquid, causing difficulties in dispersion, and would thus require larger equipment. On the other hand, sieve dimensions and the quantity of material available will set a limitation on the size of fine particles. Therefore, in practice, the coarser particles of suspended sediment (diameter greater than 0.25 mm) and the coarser particles of bed load and bed material (diameter greater than 0.5 mm) are analysed by sieving and all the finer material by sedimentation techniques. This may result in a small abrupt break in the particle size distribution curve, which may be adjusted by the use of the following approximate relationship between the diameters:

$$D_{sd} = 0.94 D_{sa} = 0.67 D_{pd}$$

where

D_{sd} = sedimentation diameter,

D_{sa} = sieve diameter, and

D_{pd} = projected diameter.

8.3 Expression of Particle Size Distribution

8.3.1 Frequency Distribution Tables

Frequency distribution tables should be prepared to present the data from size analyses in an orderly form, in the following manner:

- The total range of sizes (diameters in millimetres) should be divided into intervals, called class intervals, the number of which will depend on the classes into which the sample has been divided.
- The percentage of the total mass of the sample falling within one of these intervals will be tabulated against the interval. For example, if an interval has limits of 0.10 mm and 0.08 mm, the percentage of the total mass of the sample falling within this size range is tabulated against the interval and called the frequency of that particular class interval.
- For size distribution of coarser material, particularly for the analysis of bed load or bed material, the distribution is obtained with unequal class intervals, but for the size distribution of suspended material, a class interval of 0.02 mm is adopted over a range of 0.062 mm to 0.50 mm.
- Particles larger than 0.50 mm and smaller than 0.062 mm are broadly classified as 'Class >0.50 mm' and 'Class <0.062 mm' respectively.

8.3.2 Graphical Presentation

8.3.2.1 The data from a particle size analysis may be presented in three different graphical forms:

- Histograms,
- Frequency polygons and frequency curves, and
- Cumulative curves or particle size summation curves.

The simplest manner of depicting the results of mechanical analysis is to prepare a histogram of the data. The diameter, in millimetre is usually chosen as the independent variable, with the frequency as the dependent variable. In general, the class intervals are the abscissa, and above each class, a vertical rectangle of width equal to the class interval and height proportional to the frequency in the class is drawn (see Fig. 1).

8.3.2.2 In addition to the use of histogram as frequency diagrams, a common statistical device is to indicate variations in frequency by means of a line diagram

instead of rectangular blocks. Such frequency diagrams are called frequency polygons (see Fig. 2).

8.3.2.3 Cumulative frequency curves readily yield numerical values that serve to describe the properties of the sample in terms of size distribution. They are based on the frequency analysis of particle sizes, and drawn by choosing a size scale along the horizontal axis, and a frequency scale from 0 to 100 percent along the vertical axis. Either an arithmetic or logarithmic scale can be used for size. However, the commonest approach is to use a logarithmic scale. In practice, cumulative curves are constructed by plotting ordinates, which represent the total amount of material larger or smaller than a given diameter. Two types of cumulative curves are possible that is the more than curve, and the less than curve. Either may be used, as they provide the same type of information. Figure 3 gives an example of a more than cumulative curve.

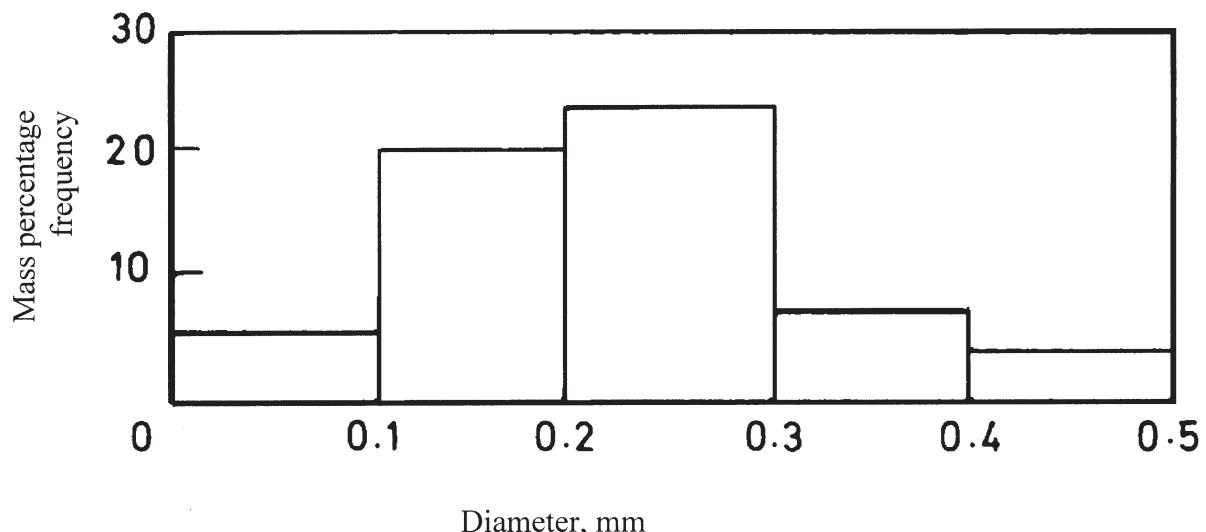


FIG. 1 EXAMPLE OF A HISTOGRAM FOR PRESENTATION OF PARTICLE SIZE DISTRIBUTION

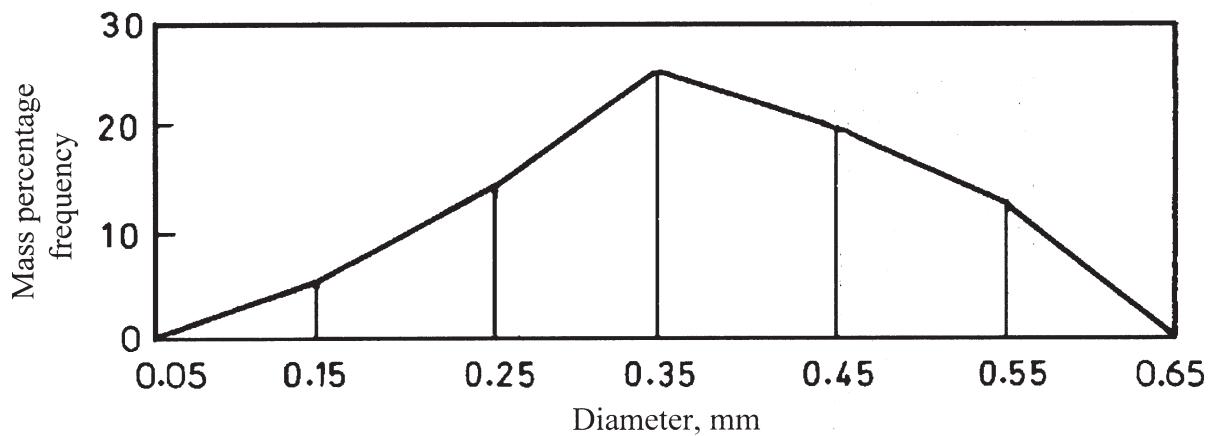


FIG. 2 EXAMPLE OF A FREQUENCY POLYGON FOR PRESENTATION OF PARTICLE SIZE DISTRIBUTION

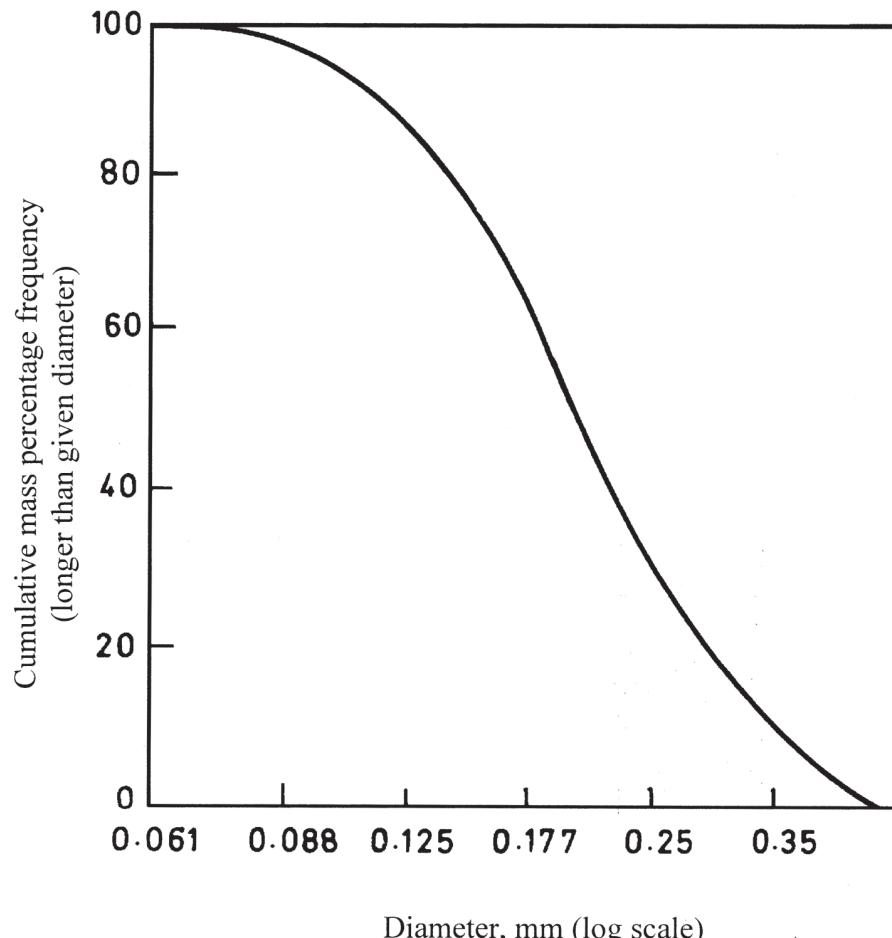


FIG. 3 EXAMPLE OF A CUMULATIVE FREQUENCY CURVE FOR PRESENTATION OF PARTICLE SIZE DISTRIBUTION

8.3.3 Basic Distribution of Bed Material

Size distribution of bed material more or less follows logarithmic normal or log normal distribution, that is the logarithm of the variable is distributed normally.

Differentiation of the cumulative distribution function leads to:

$$P(x)dx = \mathcal{O}(u) du = \frac{1}{\sigma\sqrt{2\pi}} \exp\left[-\left\{\frac{(\log x - \log \varepsilon)^2}{2\sigma^2}\right\}\right] d(\log x)$$

According to the definition:

$$\text{Mean } (\log x) = \log \varepsilon$$

$$\text{Variance } (\log x) = \text{Mean } \{(\log x - \log \varepsilon)^2\} = \sigma^2$$

Thus, the symbol ε does not denote the mean of the

variate x , but ε is defined by $\log \varepsilon$ being the mean of $\log x$.

NOTE — The variate x in this case is 'D', the particle size diameter.

9 DETERMINATION OF RELATIVE DENSITY

The method for determination of the relative density is specified in Annex F.

10 DETERMINATION OF PARTICLE SIZE DISTRIBUTION CHARACTERISTICS

The method for determination of the particle size distribution characteristics is specified in Annex G.

11 DATA PROCESSING

For both manual and automatic data processing, systematic forms and procedures are required, according to specific needs.

ANNEX A

(Clause 7.1.2)

DETERMINATION OF THE CONCENTRATION OF SUSPENDED SEDIMENT BY THE
EVAPORATION METHOD (FOR COARSE SEDIMENT PARTICLES)

A-1 PROCEDURE

A-1.1 Determine the volume of the sample and the total mass of the sample (sediment + water) plus bottle (capacity usually not less than 1 litre. However, in some countries smaller sample size is used) to the nearest 0.5 g. Record this mass as the gross mass.

A-1.2 Allow the sample to stand undisturbed so that the sediment settles out from the suspension.

Decant the sediment-free liquid after it visibly appears to be clear.

A-1.3 Wash the remaining sediment from the bottle, by means of a stream of gravity fed deionized or distilled water from a wash-bottle, into a previously weighed dry evaporating dish. Loosen the sediment adhering to the sides of the bottle by means of a rubber-tipped glass rod, ensuring that there is no loss of material during this process.

Determine the mass of the empty bottle after drying, using the same balance, and record this as the tare mass.

A-1.4 Dry the sample in the evaporating dish on a steam bath or in a drying oven. If necessary, add successive sample portions to the same dish after evaporation. The sample should be dried initially at 85 to 95°C to prevent splattering and loss of sample. After it appears dry, then dry evaporated sample for at least one hour in an oven at 101 to 105°C, cool the dish in a desiccator to balance temperature, and weigh quickly. Repeat cycle of drying, cooling, and weighing until a constant mass is obtained that is until loss of mass is less than 1 mg. Otherwise dry the evaporated sample overnight at 101 to 105°C, cool the dish in a desiccator to balance temperature, and weigh the dish and contents quickly to the nearest 0.001g.

A-2 EXPRESSION OF RESULTS

Calculate the concentration of suspended sediment, determining the concentration in relation to the mass, or the volume, of the suspension, from the formula:

$$\frac{m_4 - m_3}{m_2 - m_1}$$

or

$$\frac{m_4 - m_3}{V}$$

where

m_1 = tare mass of the bottle, in g;

m_2 = gross mass of the bottle plus sample, in g;

m_3 = mass of the empty evaporating dish, in g;

m_4 = mass of the evaporating dish plus dried sediment, in g; and

V = volume of the sample, in cm³.

Express the result in appropriate unit and state mass/volume (m/v) or mass/mass (m/m) concentration, whichever method has been used.

A-3 LIMITATIONS ON METHOD

This method is satisfactory if dealing with coarse sediment particles. With finer grained sediments, the settling time increases until a point is reached at which the method becomes impractical. Also, it becomes undesirable to decant liquid from a point close to the top of the deposited material, because of the danger of withdrawing some of the extremely fine particles. The transfer of sediment to an evaporating dish becomes difficult owing to the relatively large volume of water used for washing, which may necessitate another settling period. The use of flocculating agents will reduce the settling time, but this introduces additional material and hence requires a correction factor, which is difficult to assess with precision in routine estimates.

This method is not accurate enough for samples containing high concentration of dissolved salt. Mass of dissolved salt contributes additional mass, which may be significant especially for samples containing low concentration of suspended sediment.

ANNEX B

(Clause 7.1.3.1)

DETERMINATION OF THE CONCENTRATION OF SUSPENDED SEDIMENT BY THE FILTRATION METHOD USING FILTER PAPER

B-1 PROCEDURE

B-1.1 Exclude large floating particles (floating plants, wastes, etc) from the sample if their inclusion is not desired. For better accuracy choose sample volume to yield between 25 mg and 200 mg dried sediment in the final result. Determine the volume of the sample and the total mass of the sample (sediment + water) plus bottle to the nearest 0.5 g. Record this mass as the gross mass.

B-1.2 Selection of Filter Paper

Acid-washed, hard finished filter paper sufficiently retentive for fine particles, like Whatman No. 42 filter paper depending upon the requirement (average pore size 1.1 μm) can be used. However, for a sample which do not contain a significant quantity of fine particles, Whatman No. 40 or 41 filter paper (average pore size 2.4 μm and 4.1 μm respectively), which are faster speed filter papers can be used. While filtering through Whatman No. 40 or 41 filter papers, if the filtrate looks turbid, indicating fine particles passing through the paper, then Whatman No. 42 should be used. The rate of filtration through Whatman No. 42 filter paper is slow and hence should not be used for gelatinous sediment and need not be used for coarse sediment.

B-1.3 Dry the filter paper overnight in an oven at 101 to 105°C, cool in a desiccator to bring it to room temperature and weigh quickly (to avoid absorption of moisture from atmosphere) to the nearest 0.001 g and record it as the tare mass of the filter paper.

Fit the filter paper into a funnel. To fit it properly moisten it with a little water, press it down to the sides of the funnel either with the clean forefinger or with a flattened glass rod. Place the funnel, containing properly fitted filter paper, in a funnel stand or support it vertically in some other way. Place a clean beaker or other transparent container below the stem of the funnel to collect the filtrate. For conditions of weighing refer **A-1.4**.

B-1.4 Allow the sample to stand undisturbed for a considerable period of time and then pour the top portion of the water sediment mixture into the funnel, allowing the water to percolate. To direct the water-sediment mixture into the funnel pour the mixture down a glass rod onto the filter paper, directing the liquid against the side of the filter paper; the lower end of the glass rod should be very close to, but shall

not quite touch the filter paper. The paper is never filled completely, the level of the liquid should not rise closer than to within 5-10 mm from the top of the paper.

Slowly pour most of the water part of the sample onto the filter paper. Allow the water to percolate normally. After the water has passed through the filter paper, wash the sediment adhering to the inside of the sample bottle onto the filter paper by means of a jet of distilled (or deionized) water, from a wash bottle. If necessary, loosen the firmly adhered sediment by means of a rubber-tipped glass rod.

Weigh the empty sample bottle after drying, and record it as the tare mass.

B-1.5 To remove balance dissolved salt, if any, wash the contents on the filter paper with small portions of gravity fed deionized or distilled water from a wash bottle. Direct the water jet as near the top of the filter paper as possible and then gradually towards vertex of the cone. Let each portion of the wash water run through before adding the next. Continue washing until about 5 ml of the wash water gives no opalescence with a drop or two silver nitrate solution.

B-1.6 After all the water has passed through the filter paper, fold and place the paper in a dry evaporating dish.

Dry the contents of the evaporating dish on a steam-bath or in a drying oven at about 85-95°C until all visible water is lost, then heat to 101 to 105°C in an oven for overnight.

Remove the evaporating dish and its contents from the oven and place in a desiccator to cool to room temperature.

Weigh quickly the filter paper with dried sediment to the nearest 0.001 g taking care that no sediment particle is lost from the filter paper while taking it out of the evaporating dish and weighing. Record this as gross mass of the filter paper plus dried sediment. Make blank correction. For conditions of weighing refer **A-1.4**.

B-1.7 For blank correction, fit another identical size dried and weighed (initial mass) filter paper in a funnel. Pass the same volume of distilled water (as that of the sample volume) through it. Dry the filter paper overnight at 101 to 105°C (see **B-1.6**), cool in a desiccator and weigh. Calculate any loss in mass of the filter paper due to the passing of water through it from its initial mass minus final mass. Add the loss in mass as blank correction.

B-2 EXPRESSION OF RESULTS

Calculate the concentration of suspended sediment, determining the concentration in relation to the mass, or the volume, of the suspension, from the formula:

$$\frac{(m_4 - m_3) + (m_5 - m_6)}{m_2 - m_1}$$

or

$$\frac{(m_4 - m_3) + (m_5 - m_6)}{V}$$

where

m_1 = tare mass of the bottle, in g;

m_2 = gross mass of the bottle plus sample, in g;

m_3 = tare mass of the dry filter paper used to filter the sample, in g;

m_4 = gross mass of the filter paper with dried sediment, in g;

m_5 = initial mass of the dry filter paper used for blank correction purpose, in g;

m_6 = final mass of the dried filter paper used for blank correction purpose, in g; and

V = volume of the sample, in cm^3 .

Express the result in the appropriate unit.

B-3 RECOMMENDED PRECAUTIONS FOR THIS PROCEDURE

B-3.1 If the water sediment mixture is of such a large volume that all of it cannot be poured in at the start of the filtration, it is necessary either to add it intermittently or to arrange the apparatus so that it filters automatically. This may be accomplished by having the bottle which contains the sample inverted with its opening at the desired water elevation in the filter.

B-3.2 For coarse sediment allow the sample to stand undisturbed before filtration so that the sediment settles out from the suspension. Decant off the sediment-free liquid portion after it visibly appears to be clear. Discard the decanted water and filter the settled sediment as **B-1.4**. This will reduce filtration time. For samples containing significant proportion of fine particles or having very low sediment concentration, the entire sample should be filtered without sedimentation and decantation.

ANNEX C

(*Clauses 7.1.3.2, 7.1.3.3 and 7.1.3.4*)

DETERMINATION OF THE CONCENTRATION OF SUSPENDED SEDIMENT BY THE FILTRATION METHOD USING GOOCH OR OTHER CRUCIBLES**C-1 PROCEDURE**

C-1.1 For better accuracy choose sample volume to yield between 25 mg and 200 mg dried sediment. Exclude large floating particles (floating plants, wastes, etc) if their inclusion is not desired in the final result. Determine the volume of the sample and the total mass of the sample (sediment + water) plus bottle to the nearest 0.5 g. Record this as the gross mass.

C-1.2 Allow the sample to stand undisturbed for a considerable period of time so that the sediment settles out from the suspension. Decant as much of the sediment-free water as possible, if it visibly appears to be clear, without disturbing the sediment. Discard the decanted water.

Wash the remaining water-sediment mixture into a beaker, using distilled or deionized water. Loosen the firmly adhered sediment particles, if any, by means of a rubber-tipped glass rod and transfer all the sediment particles into the beaker.

Determine the mass of the dry empty sample bottle to the nearest 0.5 g. Record this mass as the tare mass of the bottle.

NOTE — Transferring samples to a bigger or secondary container may result in a loss of some of the samples.

C-1.3 Allow the sediment to settle in the beaker, then, if possible, decant again.

C-1.4 Prepare and insert the appropriate filtering medium. Insert a circular micro-fibre glass filter and pre-filter discs firmly over the fritted base of the crucible. To prepare glass-fibre filter disc, insert the disc with wrinkled side up into a 25 ml to 40 ml capacity Coors or Gooch crucible with adapter.

If crucibles fitted with permanent porous plates are used, such as fritted glass or alundum crucibles then no preparation of filter-mat is required.

C-1.5 Apply vacuum and wash the disc with three successive 20 ml volume of distilled water. Continue

suction to remove all traces of water. Discard washings. Remove the crucible and filter combination. Dry in an oven at 101°C to 105°C for 1 h. Cool in a desiccator to balance temperature and weigh quickly and determine mass of the crucible. Repeat cycle of drying, cooling and weighing until a constant mass is obtained or until loss in mass is less than 1 mg between successive weighing. Record the mass to the nearest 0.001 g. Store the crucible with filter in a desiccator until needed.

C-1.6 Place the crucible with filter in position in the vacuum system and begin suction. If required, wet the filter with a small volume of distilled water to seat it. Transfer the water-sediment mixture quantitatively from the beaker into the crucible, wash the beaker with distilled or deionised water, and take all the particles with washings into the crucible. Allow the mixture to filter.

To remove dissolved salts, if any, wash the contents of the crucible with small portions of distilled or deionized water from a wash bottle. Let each portion of the wash water run through before adding the next. Continue washing until 5 ml of the wash water gives no opalescence with a drop or two silver nitrate solution.

C-1.7 When filtration is complete, dry the crucible and contents at 101°C to 105°C for at least 1h. Cool in a desiccator and weigh.

Repeat cycle of drying and cooling in desiccator, and weighing until a constant mass is obtained. Weigh the crucible and contents to the nearest 0.001 g.

Record this mass as the gross mass of the crucible, filtering medium and dried sediment.

C-2 EXPRESSION OF RESULTS

Calculate the concentration of suspended sediment, determining the concentration in relation to the mass, or the volume, of the suspension, from the formula:

$$\frac{m_4 - m_3}{m_2 - m_1}$$

or

$$\frac{m_4 - m_3}{V}$$

where

m_1 = tare mass of the bottle, in g;

m_2 = gross mass of the bottle plus sample, in g;

m_3 = tare mass of the crucible with filtering medium, in g;

m_4 = gross mass of the crucible with filtering medium and dried sediment, in g; and

V = volume of the sample, in cm³.

Express the results in the appropriate units.

C-3 RECOMMENDED PRECAUTIONS FOR THIS PROCEDURE

For low concentration of solid or if the sample contains significant proportion of fine particles, which do not settle quickly, the entire sample should be filtered without sedimentation.

ANNEX D

(Clauses 7.1.3.4 and 8.1)

DETERMINATION OF THE PARTICLE SIZE DISTRIBUTION OF SUSPENDED SEDIMENT

D-1 HIGH SEDIMENT CONCENTRATIONS

D-1.1 Estimation of Total Suspended Load — Hydrometer Method

D-1.1.1 General Procedure

If the sediment concentration is high, the total suspended load can be measured with sufficient accuracy by using a hydrometer. The main advantage of this method is rapidity.

After determining the mass and volume of the sample, vigorously stir it for a few seconds in the original container using a 'figure-of-eight' motion in order to

obtain a homogeneous mixture. Introduce the hydrometer into the mixed sample immediately after stopping stirring and take the reading as soon as possible. The time after which the reading should be taken depends on the temperature of the mixed sample (see Table 1).

D-1.1.2 Use of the Hydrometer

A hydrometer is illustrated in Fig. 4. The depth to which the hydrometer is immersed gives the density of the sediment mixture, including dissolved materials. Before taking hydrometric readings, the hydrometer should be pushed gently down by about 1 mm or 2 mm

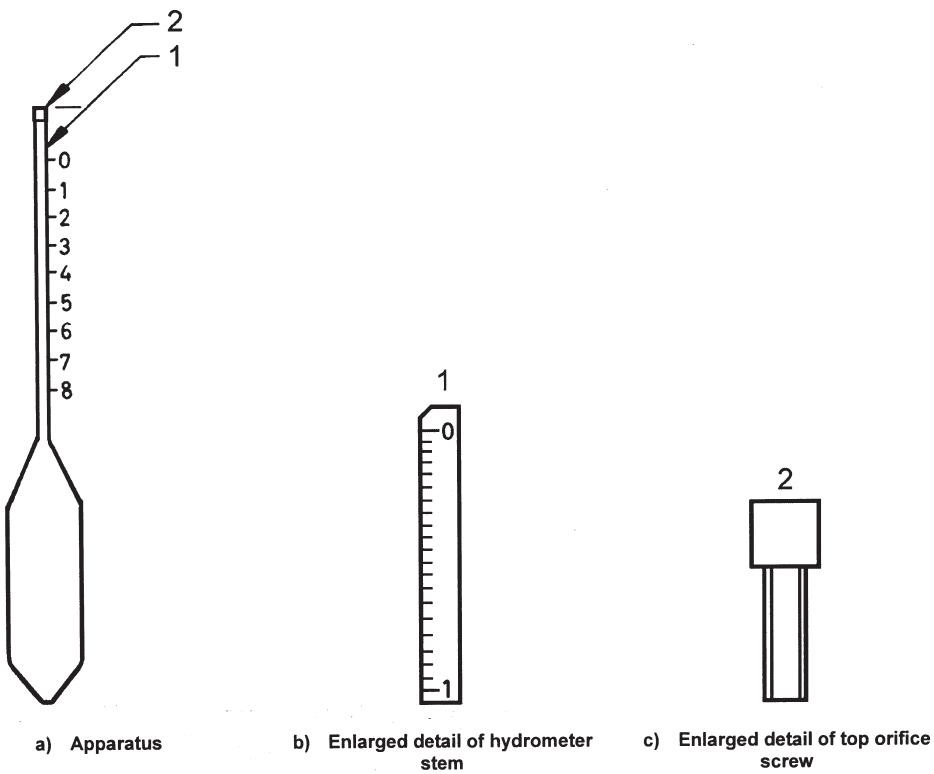


FIG. 4 HYDROMETER

Table 1 Time After which Hydrometer Readings are to be Taken in Relation to Temperature of the Sample

(Clause D-1.1.1)

Sl No. (1)	Temperature (°C) (2)	Time (s) (3)
i)	5	153
ii)	6-10	146
iii)	11-15	127
iv)	16-20	112
v)	21-25	102
vi)	26-30	92
vii)	31-35	84
viii)	36-40	77
ix)	41-45	70
x)	46-50	64

NOTE — From the mass and volume of the sample, the total concentration of suspended load and dissolved matter can be computed from the density read on the hydrometer.

and allowed to return slowly to the equilibrium position.

The hydrometer is usually calibrated for 19.4 °C and hence, should be calibrated before use in distilled water, and a chart or temperature correction curve should be prepared for readings of the hydrometer at different temperatures in distilled water.

The quantity of the total sediment in suspension plus dissolved material, in grams per litre, is given by the formula:

$$C_F + C_d = (R' - R) \times K$$

where

C_F = suspended sediment concentration, in g/litre;

C_d = concentration of dissolved matter, in g/litre;

R' = hydrometric reading of the sample;

R = hydrometric reading of distilled water at the same temperature, obtained from the calibration chart; and

K = hydrometer factor for converting the readings to gram per litre which can be determined with a salt solution of known concentration.

D-1.2 Estimation of Coarse Sediment

Collect a sufficient volume of sample (normally 5 litre) for analysis and determine its mass and volume. Pass the entire sample, including water and sediment, through a sieve of aperture size 250 µm into a container of suitable size. Wash the original sample container with distilled water to ensure complete removal of sediment. Wash the sediment retained on the sieve

thoroughly by means of a jet of distilled water until all the particles of medium and fine sediments have been washed through the sieve and into the container. Wash and dry the particles retained on the sieve and weigh. Determine the concentration, in gram per litre.

D-1.3 Estimation of Medium Sediment

Add gravity fed deionised or distilled water to the sediment collected in the container to make up the depth to 10 cm. Stir for a few seconds using a 'figure-of-eight' motion and allow to stand for the required time according to the temperature of the water as indicated in Table 2.

Table 2 Time Taken for Particles of Diameter Greater than 0.062 mm to Fall Through 10 cm Column of Water at Different Temperatures

Sl No.	Temperature °C	Time s
i)	2	45
ii)	6 to 10	40
iii)	11 to 15	35
iv)	16 to 20	31
v)	21 to 25	27
vi)	26 to 30	24
vii)	31 to 35	21
viii)	36 to 40	19
ix)	41 to 45	17
x)	46 to 50	15

NOTE — This table is not applicable for samples containing light porous material or heavy mineral particles.

Pour the supernatant water into a covered container. Repeat this process of separation of sediment by decantation of supernatant liquid followed by filling the container up to the specified height with clear water until the supernatant water is completely clear. Dry the sediment, which has settled in the beaker, after the final washing and decantation process, in an oven and weigh. Determine the concentration in gram per litre.

D-1.4 Estimation of Fine Sediment and Dissolved Matter

Calculate the estimated fine sediment and dissolved matter content, C_f , in g/litre, from the formula:

$$C_f = C_t - (C_c + C_m)$$

where

C_t = estimated total sediment concentration, per litre, determined in D-1.1;

C_c = estimated coarse sediment concentration, per litre, determined in D-1.2; and

C_m = estimated medium sediment concentration, per litre, determined in D-1.3.

D-2 LOW SEDIMENT CONCENTRATION

D-2.1 Estimation of Coarse Sediment

Proceed as described in D-1.2.

D-2.2 Estimation of Medium Sediment

Proceed as described in D-1.3.

D-2.3 Estimation of Fine Sediment — Gravimetric Method

D-2.3.1 If the sediment concentration is low, its estimation by the hydrometer method is difficult and gives inaccurate results. In such cases, estimation of total sediment is first carried out analytically by the gravimetric method (see D-2.3.2). Coarse and medium sediments are then deducted from the total mass of sediment to obtain a fine sediment fraction. Alternatively, where only the fine sediment is required, instead of analysing the total sample of sediment by the gravimetric method, it is easier to separate the coarse sediment by sieving (see D-1.2) and the medium sediment by decantation (see D-1.3) from the total sample before analysing the remaining sediment for estimation of fine sediment only by the gravimetric method.

D-2.3.2 Procedure to carry out the gravimetric method is to allow a sample of 4 litre to 5 litre to remain undisturbed in a covered container so that the sediment settles. For rapid settling of the sediment, add 2 ml to 3 ml of a 1 percent to 5 percent (v/v) alum solution depending upon concentration of the sediment. When all the particles have settled, remove the supernatant water by careful siphoning and transfer the sediment from the container to a small beaker by means of gravity fed deionized or distilled water from a wash bottle, taking care that no material is lost during this process. Carefully transfer the contents of the beaker on to a filter paper fitted in a funnel, again by means of gravity fed deionized or distilled water from a wash bottle. Wash the residue on the filter paper a few times to remove the alum. When the water has completely drained, dry the sample, on a sand bath, to constant mass. Take care to remove the material adhering to the filter paper by means of a fine brush so that no material is lost while weighing.

Deduct the concentrations of coarse and medium sediments determined as indicated in D-1.2 and D-1.3, from the total sediment concentration to obtain the concentration of fine sediment.

ANNEX E

(Clause 8.2)

DETERMINATION OF THE PARTICLE SIZE DISTRIBUTION OF
BED LOAD AND BED MATERIAL**E-1 PRINCIPLE**

Washing the sample (or dry sieving, in the case of a dry sample) through a 0.50 mm mesh sieve. Analysis of the portion retained on the sieve by drying and sieving through a further series of sieves.

Analysis of the material passing through the sieve by appropriate techniques.

**E-2 SIEVE ANALYSIS OF PARTICLES OF
DIAMETER LARGER THAN 0.5 mm**

E-2.1 In order to ensure that the data obtained by sieve analysis is comparable, the following items should be standardized, in advance, for all samples:

- a) Size of the test sample (*see E-2.2*);
- b) Type of sieving (wet or dry) (*see E-2.3*);
- c) Size of the sieves (*see E-2.4*);
- d) Method of shaking (*see E-2.5*); and
- e) Duration of sieving or end-point (*see E-2.6*).

E-2.2 The ideal quantity of sample to be used is one that covers each sieve not more than one particle deep; this implies that the mass of sample should be reduced with the smaller sieves. On the other hand, a sufficiently large sample has to be used to eliminate errors in sampling (*see IS 15360*) and weighing. In practice, the size of the sample depends upon the diameter of the coarsest particles that constitute the routine samples received by the laboratory. The mass of sample on the 200 mm diameter sieves should be 100-150 g for coarse and 40-60 g for fine sand.

E-2.3 Material may be sieved either wet or dry. While wet sieving has the apparent advantage of separating particles finer than sieve openings by washing, dry sieving is to be preferred since, in wet sieving, the formation of a water film on the screen and on the particles does not allow complete separation.

E-2.4 A set of sieves with square mesh openings, the sizes of which vary in a consistent manner, is normally used. For example, a set of sieves with square mesh openings of 4 mm, 2 mm, 1 mm and 0.5 mm, having a diameter of 200 mm and a height of 50 mm may be used. This system is arranged in a geometric series with a ratio of 2, but there are alternative systems with ratios of $\sqrt{2}$ and $4\sqrt{2}$.

E-2.5 A mechanical shaker should be used to perform sieving in one operation. With the weighed sample

placed on the mesh of the coarsest sieve, the sieves should be agitated horizontally so that the sample moves in an irregular motion over the sieves. Care should be taken to ensure that aggregates are not retained and that sieving is complete.

E-2.6 Sieving should be continued for a minimum period of 10 min or until less than 0.2 percent of the sample passes through the mesh in any 5 min sieving period. The sieving operation is carried out in 5 min stages, at the end of which the sieves are emptied and brushed in order to reduce the blocking of the apertures. The sediment fraction retained on each sieve should be carefully collected in containers and the mass of each fraction determined and recorded.

NOTES

1 Sieving is probably the easiest and most popular method of size analysis. Routinely this method is being used to determine particle size distribution of samples having particles not only larger than, even smaller than 0.5 mm, up to 0.075 mm diameter. Samples not containing a significant proportion finer than 0.075 mm diameter can be analyzed by this method. Depending upon the information to be obtained from particle size analysis, the set of sieves of desired aperture widths to be selected and used for sieving purpose.

2 There is always an element of chance as to whether a particle will or will not pass through a given sieve. Whether or not a particle will pass the sieve when it is presented at the sieving surface depends not only upon its dimension but the angle at which it is presented. Therefore as the elongation and angularity of the particles become greater the diameters obtained from sieve analysis should be viewed with caution.

3 In addition to the effects of particle shape on the validity of sieve analyses, the following sources of error, which may or may not be serious, may exist, depending upon the equipment available and the refinement of the technique:

- a) Incomplete sieving;
- b) Faulty manipulation;
- c) Sieve mesh defects; and
- d) Weighing.

Incomplete sieving is generally the most serious defect because it is not practical to sieve for a sufficient length of time to accomplish complete grade separation.

The error in sieve analysis resulting from faulty manipulation is introduced largely in the removal of the material from a sieve. Angular particles always have the tendency to become wedged in the mesh; a brush may be used to facilitate the removal of such material. Extreme care should be exercised, however, to avoid damage to the mesh or forcing the particles through the mesh.

The finer meshes should be checked frequently by using a microscope for wear or damage.

Errors in weighing, other than accidental, may be controlled by using a balance of sufficient sensitivity.

E-3 METHOD OF ANALYSIS OF PARTICLES OF DIAMETER SMALLER THAN 0.5 mm USING SEDIMENTATION TECHNIQUES

E-3.1 General

Analysis of the portion of sediment that passes through the 0.5 mm sieve may be carried out by one of the following methods:

- a) Pipette method;
- b) Hydrometer method;
- c) Siltometer method, using
 - 1) an optical level siltometer;
 - 2) a mechanical siltometer;
- d) Bottom withdrawal tube; and
- e) Visual accumulation tube.

E-3.1.1 For very fine material (diameter less than 0.062 mm), the pipette method is considered to be the most accurate method, but it is time-consuming. Within the same range, the hydrometer method, which is the simplest and most rapid, may also be used.

E-3.1.2 For grading particles with diameters in the range 0.062 to 0.50 mm, a siltometer is used. If the bulk of the material lies within this particle size range, this method gives more accurate results than sieving.

E-3.1.3 Bottom withdrawal tubes may be used over a wide range of particle sizes below 0.50 mm.

NOTE — The visual accumulation tube sand-size analyser, which is useful in the analysis of sand, that is, for the range 0.062 mm to 2 mm, may also be used.

E-3.1.4 These methods of determining particle size are based on the fact that, under the influence of gravity, a spherical particle will ultimately attain a constant velocity (settling velocity), which can be expressed by the equation:

$$C_D D^2 p Q \frac{W^2}{8} = \frac{1}{6} p D^3 g (Q_s - Q) \quad \dots(1)$$

where

C_D = drag coefficient, depending on the Reynolds number;

$Re_D = \frac{WD}{v}$ and models the shape of the particle;

D = diameter of the particle;

Q and Q_s = densities of the liquid and particle, respectively;

W = settling velocity;

g = acceleration due to gravity; and

v = kinematic viscosity of the liquid.

If the Reynolds number is small ($Re_D < 0.1$), the value

of C_D does not depend on the particle size, but only on the Reynolds number.

From $C_D = 24/Re_D$, one can proceed to Stokes' law.

$$W = 1/18 \left(\frac{Q_s - Q}{Q} \right) \times \frac{g}{v} \times D^2$$

and it can be noted that many natural sediments have a density of 2 650 kg/m³.

Equation (1) can thus be reduced to the simple relation.

$$W = f(D, C_D)$$

This relationship has been approximated in Fig. 5. C_D depends on Re_D and a shape factor F . It should be noted that for $Re_D < 0.1$, the grain diameter (D) can be defined accurately from the settling velocity W . However, for $Re_D > 0.1$, only an approximation is possible.

E-3.1.5 The pipette method and the hydrometer method are suitable for analysis of particles of sizes less than 0.062 mm, but since particles of such small sizes are generally not significant in the bed load or bed material, these methods are not used for routine analysis of such bed samples. However, in special cases where it is necessary to know the distribution of material finer than 0.062 mm, use of the pipette method is justified.

E-3.1.6 In case of use of a siltometer and bottom withdrawal tube, the sedimentation tube should be examined for possible deposits at the end of the experiment.

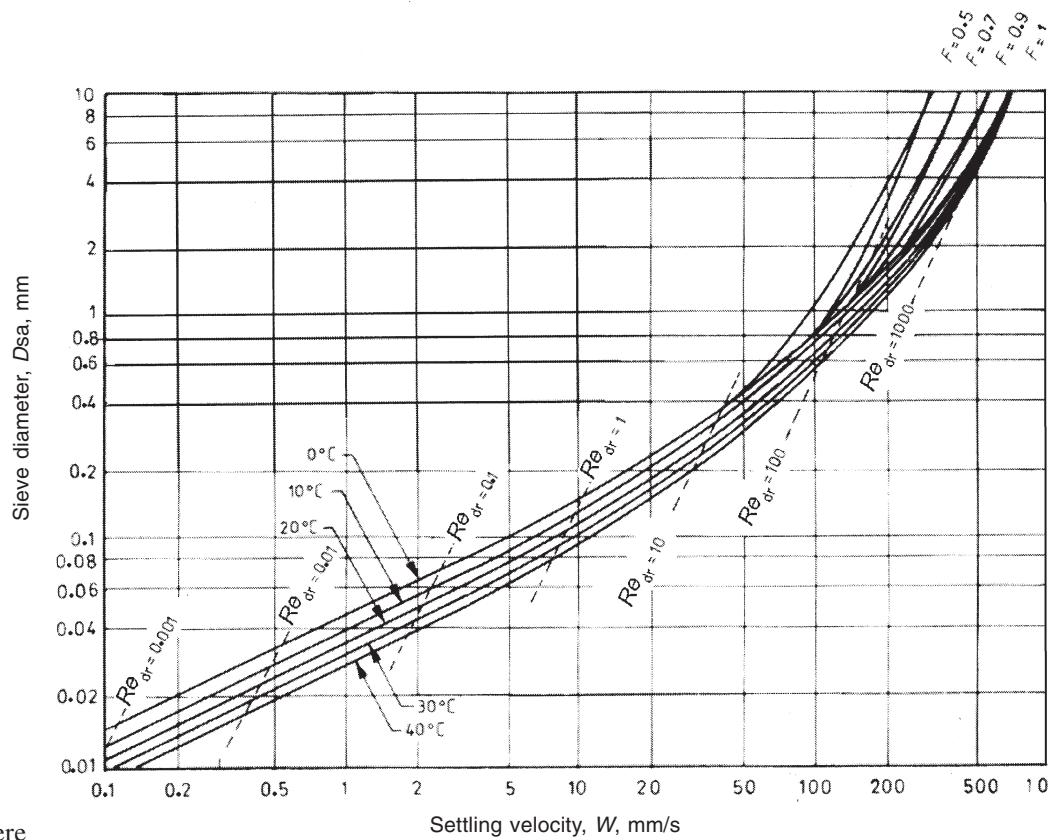
E-3.2 Siltometer Method

Siltometers work on the principle of the different settling velocities of particles in a column of liquid (usually water). They allow direct determination of the rate of accumulation of the material by measuring the sediment deposited at the bottom of the tube.

In the operation of siltometers, assumptions are made that the particles to be measured are evenly distributed at the start of the test and that they have the same relative density. Furthermore, there is an implicit assumption that the settling velocity of each particle is independent of the others, a condition which is considered applicable if the concentration of the particles is not high (up to 2 percent, although such methods can be used for concentrations up to 5 percent).

Particles of different sizes fall through the column of water at different velocities; the size distribution of the particles varies throughout the suspension and the concentration decreases as sedimentation continues.

The sample should be allowed to settle for a time sufficient to allow all particles over a certain selected



where

$$Q_s = 2650 \text{ kg/m}^3$$

$$Q = 1000 \text{ kg/m}^3$$

$$F = \text{shape factor} = \frac{c}{\sqrt{ab}}$$

where c is the minor axis, a is the major axis and b is the intermediate axis of three mutually perpendicular axes

$$Re_D = \text{Grain Reynolds number} = WD_{sa}/v$$

FIG. 5 PARTICLE DIAMETER VERSUS SETTLING VELOCITY

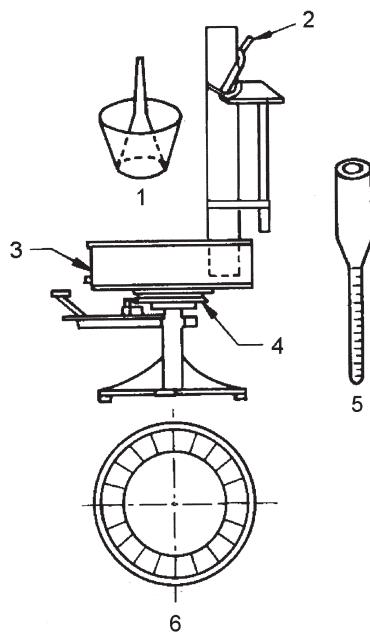
size to fall to the bottom of the column of water and the different fractions should be collected in separate bores that move into position under the column of water at predetermined intervals of time. Figure 6 illustrates the essential features of the siltometer, which consists of following four main parts:

- Sedimentation tube, made of brass tubing, of uniform inside diameter about 6 cm and 2 m long, attached to lever B so that, by actuating this lever, the entire tube may be raised through about 10 mm;
- Sedimentation dropping device A , made of two funnels, the purpose of which is to introduce the sediment under water. The funnels fit tightly together, allowing the sediment to be introduced without immediately entering the sedimentation tube. At the desired time, the inside funnel is lifted and the sediment enters the main tube;

- Sediment collecting device, consisting of a circular aluminium trough C , divided around its circumference in to 20 individual pockets. The apparatus is so constructed that this collection trough may be moved-around 1/20th of its circumference by operating a lever manually at any desired time;
- Container D , with a slender, graduated stem, used when volumetric measurement of the sediment collected in each pocket is desired.

The procedure for analysis of sediment using the mechanical (Puri's) siltometer is specified below.

Fill the circular trough with water to a depth of 100 mm, and place a rubber stopper in the pocket (this should be pocket 20) immediately below the sedimentation tube. Lower the tube by means of lever B , so that the stopper fits tightly in the end of the tube. Fill the sedimentation tube with water and insert snugly the inner funnel.

**Key**

1. Dropping device	4. Iron stop spring
2. Lever	5. Silt measuring tube
3. Aluminium trough	6. Plan of trough with collecting box

FIG. 6 ESSENTIAL FEATURES OF A MECHANICAL SILTOMETER FOR SEDIMENT (0.06 mm to 0.60 mm)

Measure the temperature of the water in the tube. Place a sample of about 10 g of sediment in the dropping device and cover with water. Separation into fractions by size may now commence. Quickly remove the inserted funnel and simultaneously start a stopwatch. Remove the remaining funnel section as rapidly as possible and tightly insert a rubber stopper into the top of the sedimentation tube. As soon as the rubber stopper has been inserted tightly into the top of the sedimentation tube and the tube has been raised by actuating lever B, immediately rotate the trough by means of the lever so that the next pocket (that is No. 1) is positioned below the tube outlet to collect the sediment. Allow sedimentation into this first cup to continue until 26 s have elapsed, then rotate the trough one division to place the second pocket (No. 2) in position. Continue rotation of the trough at 4 s intervals up to 66 s, then at 10 s intervals up to 106 s, and then after 136 s, 166 s, 196 s, 376 s and 556 s. When separation has been completed, which takes less than 10 min, filter the material in each compartment dry it and weigh.

This procedure is suitable only for sediments with particle sizes between about 0.06 mm and 0.60 mm.

Table 3 gives the deduced diameter of sediment particles settling through a vertical water column of 2 m length in various times and at different temperatures.

Table 3 Deduced Diameter in Micrometres of Sediment Particles Settling through a 2 m Vertical Container in Various Times and at Different Temperatures (After Puri with Zahm's Correction)
(Clause E-3.2)

Sl No.	Temp- erature °C	Times																				
		26	30	34	38	42	46	50	54	58	62	66	76	86	96	106	136	166	196	376	566	
(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)	(9)	(10)	(11)	(12)	(13)	(14)	(15)	(16)	(17)	(18)	(19)	(20)	(21)	(22)	
i)	10	618	541	483	438	401	371	346	325	307	291	277	249	227	209	195	165	146	131	91	74	
ii)	11	612	536	478	433	397	367	342	322	303	287	274	246	224	207	193	163	144	130	90	73	
iii)	12	606	530	473	429	393	363	339	317	300	284	271	243	221	204	190	161	142	128	88	72	
iv)	13	600	525	468	427	389	360	335	314	297	281	268	240	219	202	188	159	140	126	87	71	
v)	14	594	520	464	421	385	356	332	311	294	278	265	237	217	200	186	157	138	125	86	70	
vi)	15	588	515	459	416	381	353	328	308	290	275	262	235	214	198	184	155	137	123	85	69	
vii)	16	583	510	455	412	378	349	325	305	287	272	259	232	212	196	182	153	135	122	84	68	
viii)	17	577	55	451	408	374	346	322	302	285	270	256	230	209	193	180	152	134	120	83	67	
ix)	18	572	501	446	404	370	342	319	299	282	267	254	228	207	191	178	150	132	119	82	66	
x)	19	567	496	443	400	367	339	315	296	279	264	251	225	205	189	176	148	130	118	81	66	
xi)	20	562	492	438	397	363	336	312	293	276	262	249	223	203	187	174	147	129	116	80	65	
xii)	21	557	487	435	393	360	333	310	290	274	259	246	221	201	185	172	145	127	115	79	64	
xiii)	22	553	484	431	390	357	330	307	288	271	257	244	219	199	183	171	144	126	114	78	63	
xiv)	23	548	480	428	386	354	327	304	285	268	254	242	217	197	182	169	142	125	113	77	63	
xv)	24	544	476	424	383	351	324	302	282	266	252	240	215	195	180	167	141	124	112	76	62	
xvi)	25	540	472	421	380	348	321	299	280	264	250	237	213	193	178	166	139	122	110	76	61	
xvii)	26	535	468	417	377	345	319	296	278	261	247	235	211	192	177	164	138	121	109	75	60	
xviii)	27	531	465	414	374	342	316	294	275	259	245	233	209	190	175	163	136	120	108	74	60	
xix)	28	527	461	411	371	339	313	291	273	257	243	231	207	188	173	161	135	119	107	73	59	
xx)	29	522	458	407	368	337	311	289	271	255	241	229	205	186	172	160	134	118	106	72	59	
xxi)	30	518	454	404	365	334	308	287	268	253	239	227	203	185	170	158	133	116	105	72	58	
xxii)	31	515	451	401	362	331	306	284	266	251	237	225	202	183	169	157	131	115	104	71	57	
xxiii)	32	511	447	398	359	329	304	282	264	249	235	224	200	182	167	156	130	114	103	70	57	
xxiv)	33	507	444	395	357	326	301	280	262	247	233	222	198	180	166	154	129	113	102	70	56	
xxv)	34	503	441	392	354	324	299	278	260	245	231	220	197	179	164	153	128	112	101	69	56	
xxvi)	35	500	437	389	351	321	297	276	258	243	230	218	195	177	163	151	127	111	100	68	55	

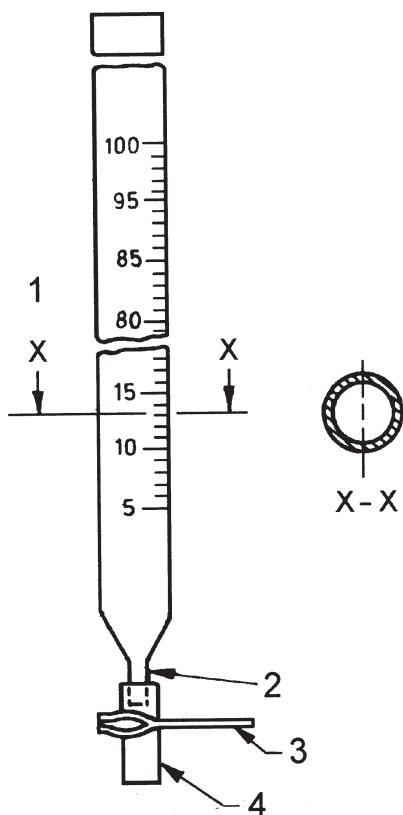
E-3.3 Bottom Withdrawal Tube Method

E-3.3.1 Bottom withdrawal tubes are widely used for analysis of sediment particles of diameter less than 0.5 mm.

They work on the Oden principle of uniform dispersion in sediment systems, which states that in a uniformly dispersed suspension (as used in the bottom withdrawal tube), the sediment concentration at any level remains constant until the largest particle in suspension has had time to settle from the surface to the level in question.

The apparatus consists of a straight glass tube (see Fig. 7), 1 m long, having a uniform diameter of 25.4 mm. There is an outlet at the bottom, which makes possible the rapid withdrawal of material accumulated at the lower end of the tube.

NOTE — The constancy of diameter of the bottom withdrawal tube should be checked and the variation should not exceed ± 1.5 percent of the main value.



Key

1. Depth, centimetres

2. Nozzle $\begin{cases} \phi_{\text{ext}} = 10 \text{ mm} \\ \phi_{\text{int}} = 7 \text{ mm} \end{cases}$

3. Pinch clamp

4. Rubber tube

FIG. 7 EXAMPLE OF A BOTTOM WITHDRAWAL TUBE

The procedure for analysis of sediment using bottom withdrawal tube method is specified below.

Uniformly disperse the sample in the bottom withdrawal tube, and then clamp in a vertical position (solid particles begin to settle at the bottom, the settling velocity being a function of the relative density and size of individual particles).

Intermittently, withdraw fractions of the settled sediment from the bottom of the tube into containers (bottles), and place in an oven controlled at 85°C to 95°C until the sediment appears dry, then dry it at 101°C to 105°C to constant mass, then cool and weigh. (Withdrawal should be carried out after periods of time of corresponding to those required for particles of given sizes to pass through the length of the water column. Each fraction is then dried and the mass of the accumulated sediment determined.)

The mass of sediment of each concentration that would remain in suspension may be computed from these data, assuming that the water column has remained at the original height. Since the height of the water column actually decreases with each withdrawal, a pro rata correction has to be made to obtain the amount of suspended material that would remain in a column of constant height. Similarly, the hypothetical settling time for each fraction in a column of constant height may also be computed. The computed percentage of material remaining in suspension at the moment of withdrawal, relative to the total amount of material present in the original sample, together with the corresponding settling time, provide the data necessary to construct an Oden curve (see Fig. 8). Extending a tangent from any point on the curve to the ordinate scale will indicate the amount of material in the sample that is finer than the particle size represented on the corresponding time abscissa at the point of tangency. Graduation of the sediment according to any desired scale of particle sizes can be determined in this manner. Since the settling velocity is sensitive to temperature and changes in the medium, variations in temperature shall be considered in correlating particle sizes and settling time.

E-3.3.2 Particular attention shall be given to the following procedures while conducting the test.

Before commencing the analysis, shake the bottom withdrawal tube for about 5 min at intervals of approximately 5 s (which is approximately the time required for an air bubble to move from one end to the other).

Take care that the stopwatch is started only when the air bubble starts upwards from the bottom of the tube in the supporting stand.

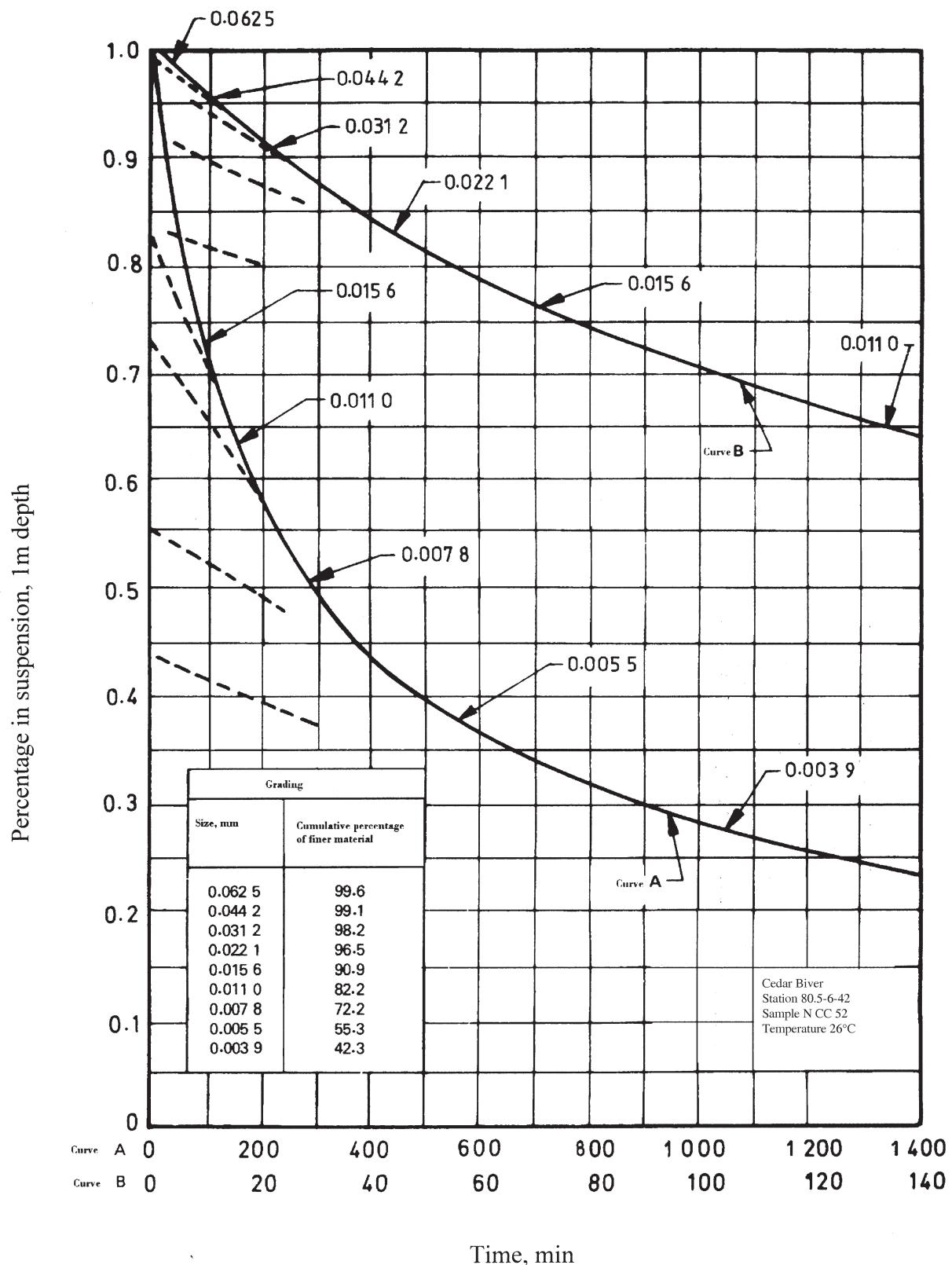


FIG. 8 EXAMPLE OF AN ODEN CURVE (CONSTANT DEPTH, VARIABLE TIME)

Determine beforehand the time after which each fraction is to be taken, considering the time required to run-off a column of liquid.

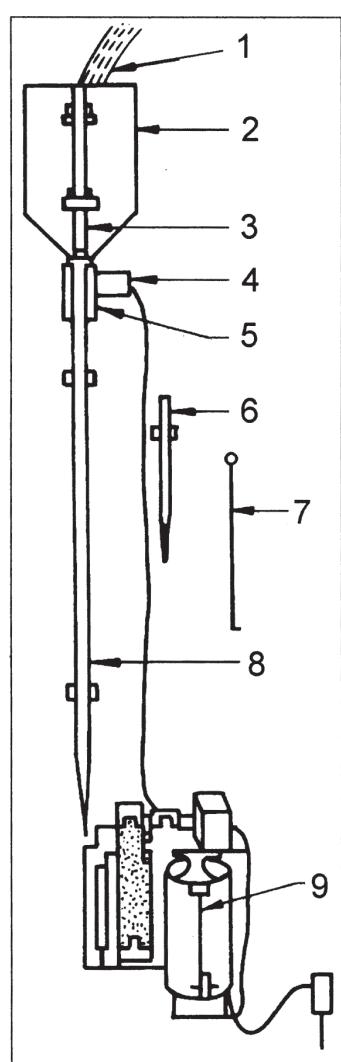
When reading the percentage distribution of particle size, draw two tangents, wherever possible, to allow counter checking.

E-3.4 Visual Accumulation Tube Method

This method is used for the analysis of sands (diameter 0.062 mm to 2 mm).

E-3.4.1 Apparatus

The apparatus consists of the following main elements (see Fig. 9).



Key

1. distilled water	6. thermometer
2. glass funnel	7. mixing rod
3. rubber tube	8. glass tube ($\phi_{int} = 25 \text{ mm}$, Min)
4. valve switch	9. recorder
5. valve mechanism	

FIG. 9 VISUAL ACCUMULATION TUBE

E-3.4.1.1 Glass funnel

About 250 mm long, bearing a reference mark on the stem of the funnel indicating the proper height for the water.

E-3.4.1.2 Rubber tube connecting the funnel

The main sedimentation tube and which, together with a special clamping mechanism, serves as a valve. Details of the mechanism for clamping the rubber tube together are shown in Fig. 10.

E-3.4.1.3 Glass sedimentation tube

Tubes may be one of two lengths.

A 1 800 mm tube has a section 1 400 mm long, of 50 mm inside diameter, a 200 mm constricting section, and a 200 mm accumulation section of 10 mm inside diameter. This long tube is used for the analysis of bed, beach or other coarse sands when sufficient quantum of sample is available.

A 1 200 mm tube has a section 800 mm long, of 25 mm inside diameter, a 200 mm constricting section and a 200 mm accumulation section with an inside diameter of 2.1 mm; 3.4 mm; 5.0 mm or 7.0 mm.

The short tube is suitable for the analysis of samples that contain only small quantities of sand, most of which has a sieve diameter less than 1 mm. An elastic plug is inserted into the bottom of the accumulation section.

E-3.4.1.4 Electricity operated tapping mechanism

It strikes against the glass tube and helps keep the accumulation of sediment uniformly packed and level on top.

E-3.4.1.5 Visual accumulation tube recorder

It comprises,

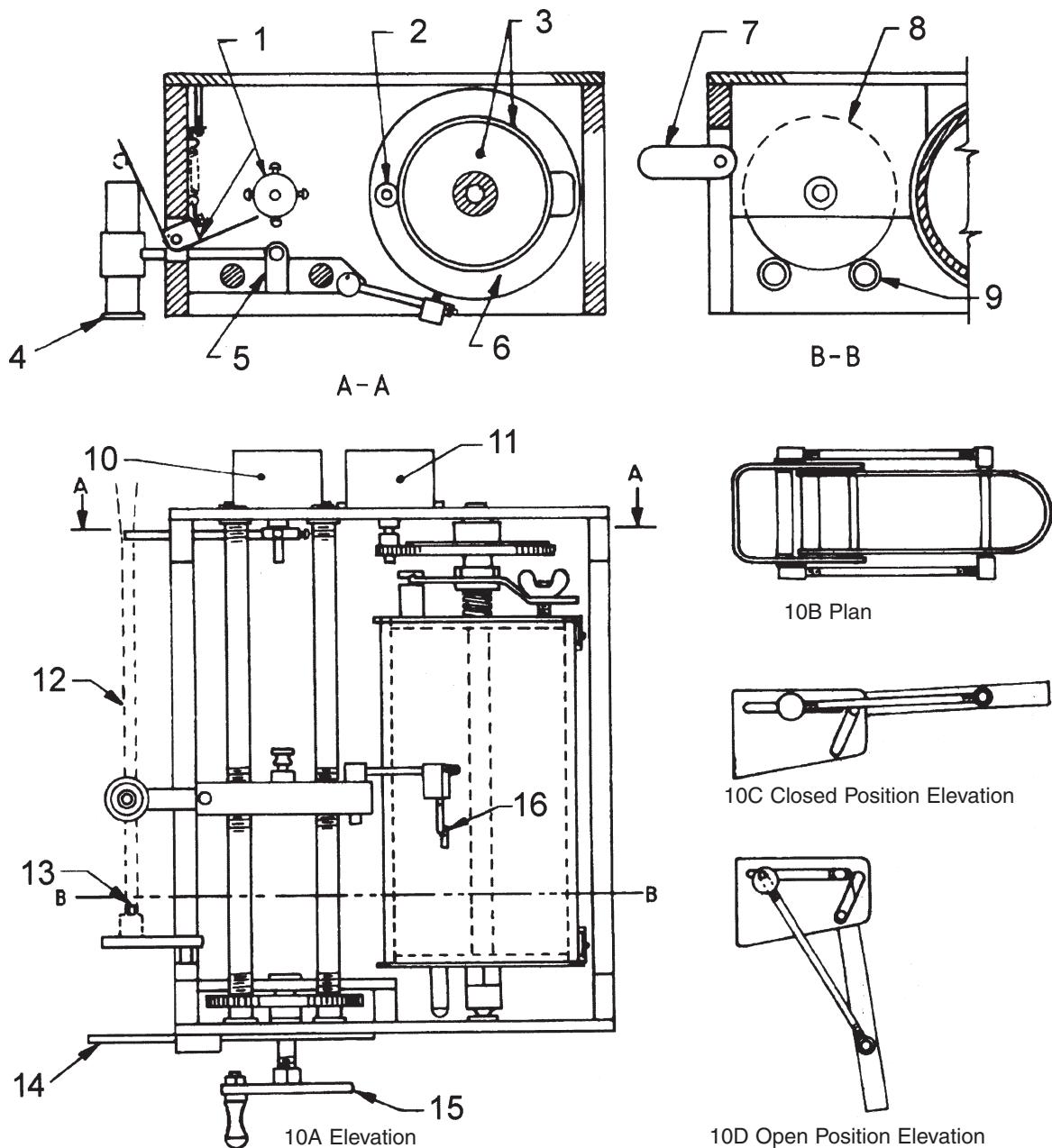
- carriage, which can be moved vertically by a hand-operated mechanism and on which are mounted a recording pen and an optical instrument consisting of a two-power telescope eyepiece with a horizontal cross hair; and
- cylinder, which carries a chart and rotates at a constant rate during the analysis.

E-3.4.1.6 Recorder chart in printed form

It incorporates the fall-diameter calibration; the recorder pen draws a continuous curve of sediment accumulation on the chart.

E-3.4.2 Samples for Analysis

Samples having particles that are mainly in the range of sand sizes are suitable for analysis by this method.

**Key**

1. Tappers	9. Small gear
2. Clock gear	10. Tapper motor
3. Gear and clutch	11. Timing motor
4. Eyepiece	12. visual tube
5. Carriage	13. Tube plug
6. Chart cylinder	14. Beaker shelf
7. Tube bracket	15. Hand wheel
8. Drive gear	16. Pen

FIG. 10 VISUAL ACCUMULATION TUBE MECHANISMS

The mass of sample may be as little as 0.050 g for fine sands, and as large as 15 g for samples with a normal particle size distribution. If many coarse particles, of sieve diameter larger than 1 mm or 2 mm, are present in a sample, they should be removed by sieving. If a large concentration of clay or silt (sizes under 0.062 mm) is present in a sample, it should be removed before analysis. Some coarse silt does not affect the accuracy of results, but appreciable quantities of silt require additional time for the analysis. The clay and silt fractions should be separated from the sand by sieving or by sedimentation processes, but the division need not be at a precise size.

NOTE — Since the calibration of the tube is carried out with sand particles, that is, the particles which are of irregular shape and for which the density for each sample is about $2\ 650\ \text{kg/m}^3$, even though many particles of higher and lower densities may sometimes be included, for special particle shapes or samples with densities different from $2\ 650\ \text{kg/m}^3$, accurate analyses may require special calibration.

E-3.4.3 Preparation of Samples for Analysis

As most samples to be analysed by this method originally contain clays and silts, the separation of sand sizes from the finer material prior to analysis is a basic problem. The more thoroughly the clays and silts are removed from the sample, the simpler and faster the analysis will be. Present methods of removing clays and silts are not entirely satisfactory, and further investigation of the problem should be carried out with a view toward reducing the total time of analysis.

Two wet-sieving processes may be used to separate sands from the finer sizes. If separation is carried out using a 0.062 5 mm sieve, some particles with sieve diameters larger than 0.062 5 mm will pass the sieve but, partly because of incomplete sieving, many particles with sieve diameters much smaller than that size will be retained on the sieve. An accurate sedimentation analysis requires that the silt (sieve diameters smaller than 0.062 5 mm) retained by the sieve be identified and also that the sand (sieve diameters coarser than 0.062 mm) be identified in the fraction passed by the sieve. If separation is carried out using a 0.050 mm sieve, very little sand will be passed and an accurate sedimentation analysis can be performed by combining sedimentation analysis of the clay and silt in the passed fraction, with the visual accumulation tube analysis of the retained fraction. However, the smaller the opening of the sieve, the more time-consuming and difficult the sieving process is.

By another process, initial separation of sands from silts and clays may be carried out using a sedimentation tube. The sample is introduced at the top of a tube and allowed to settle for the time that will permit, for the given water temperature and distance of fall, all the particles with sedimentation diameters greater than

0.062 5 mm to settle to the bottom of the column. The settled part of the sample may be analysed in the tube, and the part not settled might be analysed by any method appropriate to silts and clays. This type of separation allows accurate analyses, but the time for analysis in the tube may be undesirably long because some fine material may be present.

Sand particles should be thoroughly soaked in water before analysis so that every particle is completely wetted. They should be contained in not more than 40 ml of water at a temperature not lower than that of the water in the tube. Samples for analysis should be relatively free of organic matter and in such condition that the grains will fall as individual particles and not as aggregates.

If the organic matter in a sample is of sufficient quantity to decrease the accuracy of analysis, it will be visible in the sample and very obvious as the sample settles in the tube. Also, the presence of aggregates is noticeable through the eyepiece, and a competent operator will realize that the analysis is erroneous.

Whether particles will fall individually may be determined in a beaker prior to analysis by stirring the immersed sample in a rapid circular motion for a few moments and then allowing the particles to settle and accumulate at the bottom of the container. If there is only a slight tendency to form aggregates repeated washing by adding distilled water to the sample, stirring, allowing to settle, and decanting the supernatant liquid will generally improve the settling characteristics.

Organic matter, which may be objectionable because of its volume or because it forms a binding agent for floccules, may be oxidized by adding a 6 percent hydrogen peroxide solution to the sample contained in about 40 ml of water, using about 5 ml of solution for each gram of dry sample. Thoroughly stir and mix and cover it. If the oxidation is slow, or after it has slowed, heat the mixture to 95°C and allow it to remain at that temperature. Occasional stirring and the addition of more hydrogen peroxide, can be used until the oxidation appears complete. Then washing two or three times as described above adequately prepares the sample for analysis, except that additional cooling may be desirable.

The water in the tube should be frequently changed to avoid contamination from repeated analyses of treated samples. The contamination does not appear to alter the accuracy of the analyses but, if the sample is dried and weighed after analysis, the mass may be affected.

E-3.4.4 Selection of Tube Size

Before performing an analysis, it is necessary to choose the proper tube size for the given sample. Frequently, two sizes or more would be satisfactory.

The quantity of sand and the upper particle size limit in a sample are used as guides in selecting the tube size. Table 4 indicates the size of tube for limits of sand in samples. If the pertinent characteristics of samples are not known from previous experience of the source of the sample, the sample to be analysed may be compared with a set of synthetic samples. For example, a sample may be analysed in a 2.1 mm tube if it does not exceed, in quantity of particle size, a synthetic sample containing 0.8 g of sand with a maximum particle size of 0.250 mm.

The maximum particle sizes shown in Table 4 are those that should not be exceeded by a significant percentage of the sample. The percentage of excess may be greater if the sample is small in relation to the capacity of the tube or if the analysis of the coarser portion is not highly important.

Normally, the best results are obtained if the total height of accumulation in the bottom of the tube is between 25 mm and 100 mm. If a sample has a very limited size range, or if the material is predominately coarse better results are obtained with maximum heights less than 100 mm. If a satisfactory tube size is not selected for the first analysis, the sample can be transferred to a tube of different size. However, the choice of a suitable tube is not difficult because the usable limits of the respective tubes overlap considerably.

Table 4 Guide to Selection of Correct Tube Size

SI No.	Sample		Maximum Particle Size		Sedimentation Tube	
	Dry Mass (1)	Volume of Sand (2)	Sedimentation Diameter mm (4)	Sieve Diameter mm (5)	Length mm (6)	Dia- meter mm (7)
i)	0.05-0.8	0.03-0.5	0.250	0.250	1 200	2.1
ii)	0.4-2.0	0.2-1.2	0.350	0.400	1 200	3.4
iii)	0.8-4.0	0.5-2.4	0.500	0.600	1 200	5.0
iv)	1.6-6.0	1.0-4.0	0.700	1.000	1 200	7.0
v)	5.0-15.0	3.0-9.0	—	2.000	1 800	10.0

E-3.4.5 Method of Analysis

Analyses may be performed in less than 10 min, if the particles in the sample have sedimentation diameters greater than 62 μm . More time is required if silt is present in the sample. The recommended procedure is as follows.

E-3.4.5.1 Choose a chart appropriate to the length of the tube and record the identity of the sample, the operator and an identification of the analysis. Place the chart on the cylinder.

Ensure that the baseline of the chart is parallel to the bottom of the cylinder so that the pen trace will be parallel to the baseline except when sediment is accumulating.

NOTE — The 1 800 mm and the 1 200 mm tubes require different charts because of the unequal distances through which the sample shall settle (see Fig. 11 and Fig. 12).

E-3.4.5.2 Set the recorder pen on the zero accumulation zero-time lines of the chart. The pen should start to the right of the zero-time line and should be brought to the line by the motor-driven rotation of the cylinder.

E-3.4.5.3 Adjust the recorder to bring the horizontal hair in the eyepiece level with the top of the tube plug where the accumulation of sediment begins.

E-3.4.5.4 When the apparatus, including the proper sedimentation section, has been assembled fill the tube with distilled water to just above the valve. Measure and record the temperature of the water in the tube, and close the valve. The water need not normally be changed after each analysis.

E-3.4.5.5 Start the electrical tapping mechanism; this operation also closes the electrical circuit to a switch at the valve so that rotation of the cylinder will start when the valve is opened.

E-3.4.5.6 Wash the sand sample into the funnel above the closed valve with gravity fed deionised or distilled water. Fill the funnel to the reference mark, and then stir the sample briskly for 10 s using a special stirring rod.

E-3.4.5.7 Immediately open the valve fully. Because opening the valve automatically starts the cylinder, the chart time and the settling of the particles in the tube begin simultaneously.

E-3.4.5.8 Look through the eyepiece and as soon as the first particles reach the bottom of the tube move the carriage vertically at a rate that keeps the horizontal hair level with the level of the accumulation of sediment. Continue until the pen has passed the 62 μm mark on the chart.

Rotation of the cylinder should automatically stop. If material is still settling, the tracking operation is continued, at least intermittently, until the maximum accumulation height is determined.

E-3.4.5.9 While the pen is at the minimum accumulation height, release the cylinder drive clutch and rotate the cylinder by hand to extend the line of maximum accumulation across the chart.

E-3.4.5.10 When the valve has closed, drain the sample into a beaker by removing the tube plug. Open the valve slightly to drain out excess water and to wash out the lower end of the tube more completely with gravity fed deionised or distilled water. Replace the plug.

E-3.4.5.11 Determine the size distribution from the chart as follows:

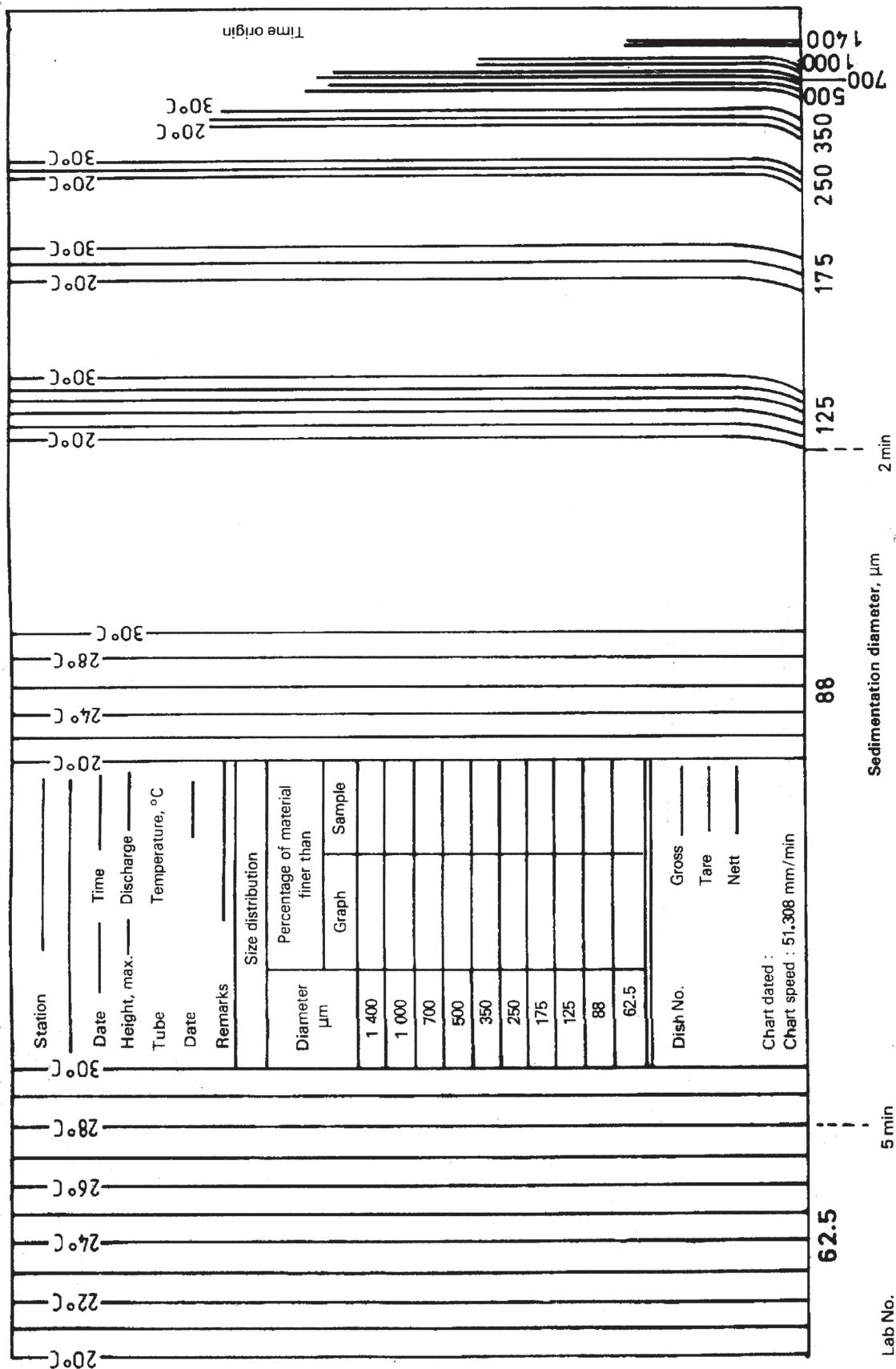


FIG. 11 CALIBRATED CHARTS FOR THE ANALYSIS OF SANDS BY THE VISUAL ACCUMULATION TUBE METHOD USING A 1800 mm TUBE

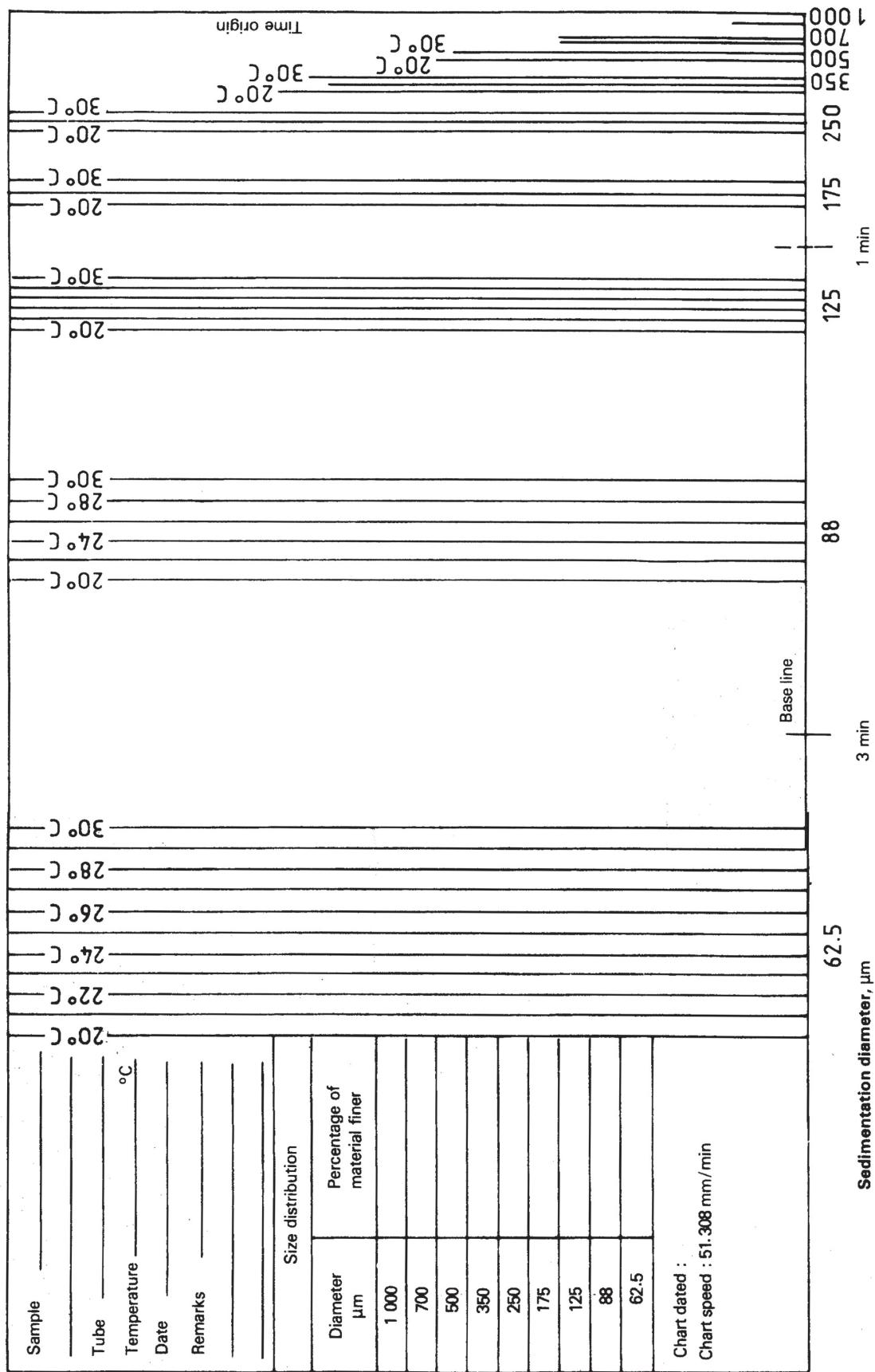


FIG. 12 CALIBRATED CHARTS FOR THE ANALYSIS OF SANDS BY THE VISUAL ACCUMULATION TUBE METHOD USING A 1200 mm TUBE

The trace should be a continuous curve of sediment accumulation with time as the abscissa and the height of accumulated sediment as the ordinate. Generally, analytical results are desired as percentages of the sample finer (or coarser) than certain definite sizes. One common series of these sizes is shown on the calibrated chart (*see* Fig. 13 and Fig. 14). The percentages finer than those sizes on the chart may be read from the chart by use of a scale that will conveniently divide the total accumulation into 100 equal parts. The intersections of the accumulation curve and the division-size lines (interpolated, if need be) for the temperature of analysis are marked by ticks. Spread the chart out flat, place the 'percent' of the scale on the total-accumulation line and the '100 percent' on the zero accumulation line. Move the scale horizontally to the intersection of the curve with the size-temperature line. If horizontal lines, instead of ticks are drawn through the intersections, all percentages may be read from one position of the scale. The percentage of material finer than the division size may be read directly on the scale as it is represented

by the portion of the total accumulation that lies above the curve. Percentage contents of coarser material may be read by reversing the scale.

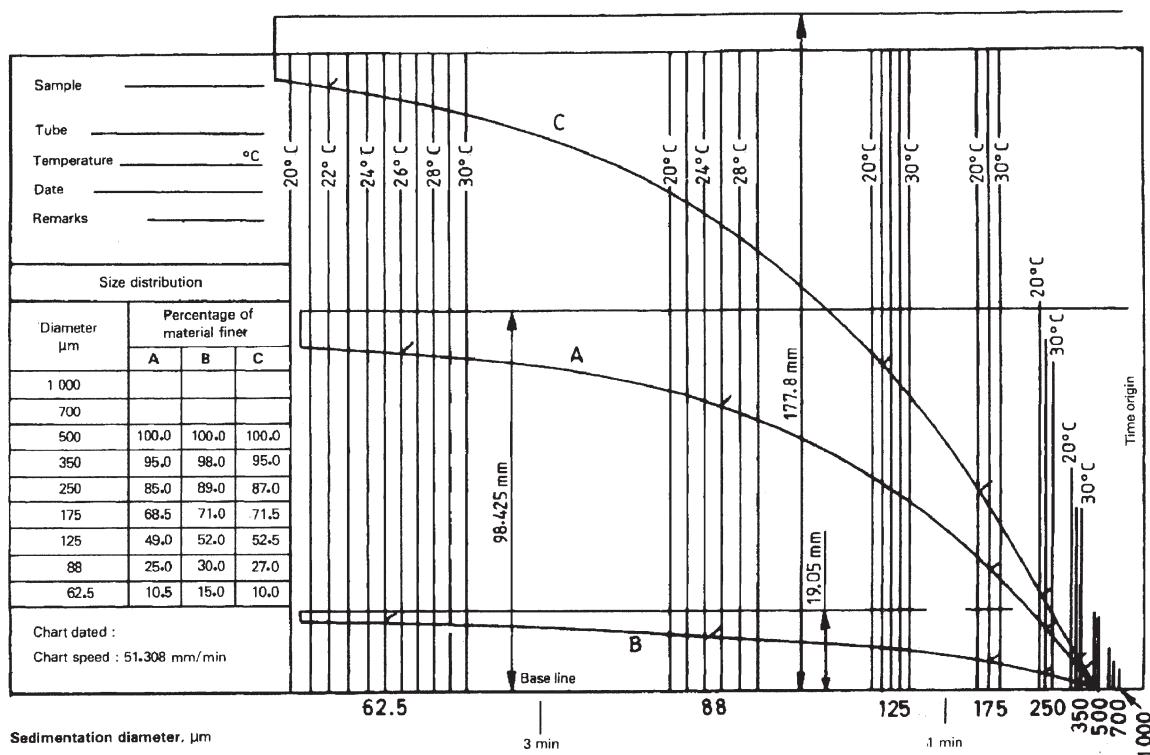
If 10 percent of material coarser than that analysed was removed from the sample before the analysis, then the 90 percent mark may be used on the zero-accumulation line to show readings directly in percentages of the total sample. Similarly, if 40 percent of the original sample was removed as silt and clay before the analysis, the 60 percent mark may be used on the total-accumulation line to obtain direct readings in percentages of the total sample.

E-3.5 Pipette Method

This method can be used for analysis of particles of diameter less than 0.06 mm.

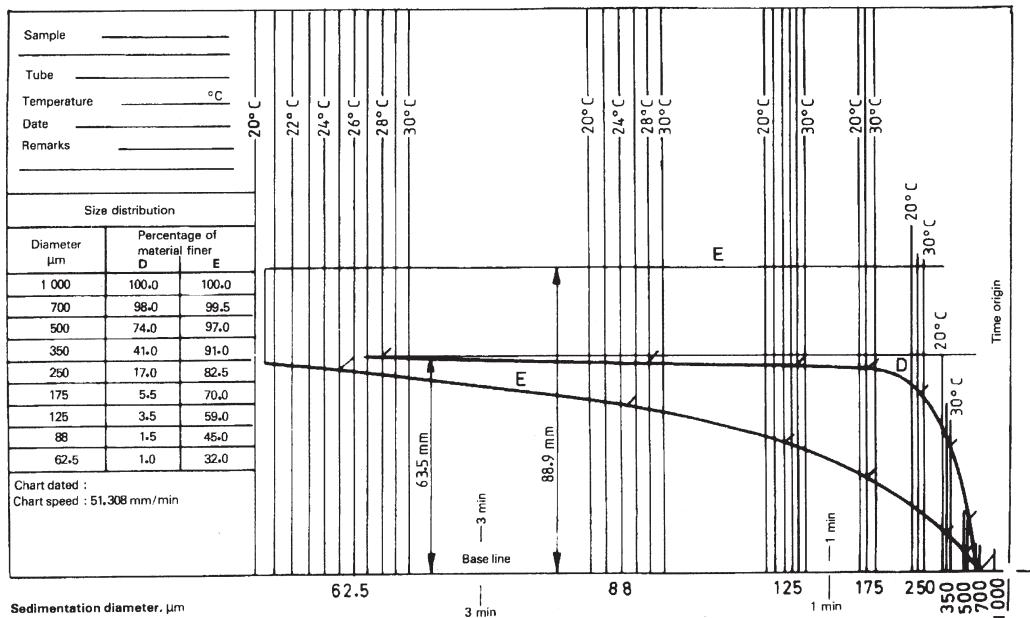
E-3.5.1 *Apparatus*

E-3.5.1.1 Vertical glass jar, about 2.5 litre to 3 litre capacity, diameter approximately 16 cm and height approximately 32 cm.



A — Powder River sand, fine distribution, sample No. 7, 5.0 mm tube, 26°C
B — Powder River sand, fine distribution, sample No. 1, 2.1 mm tube, 26°C
C — Powder River sand, fine distribution, sample No. 9, 5.0 mm tube, 26°C

FIG. 13 TYPICAL CURVES FROM A VISUAL ACCUMULATION TUBE ANALYSIS USING A 1200 mm TUBE



D — Cheyenne River sand, sample No. 4, 7.0 mm tube, 28°C

E — Powder River sand, fine distribution, sample No. 2, 2.1 mm tube, 25°C

FIG. 14 TYPICAL CURVES FROM A VISUAL ACCUMULATION TUBE ANALYSIS USING A 1200 mm TUBE

E-3.5.1.2 Sedimentation tube, narrow mouth one litre capacity measuring cylinder with air-tight stopper (or rubber cork) used as sedimentation tube.

E-3.5.1.3 Evaporating dishes, porcelain dishes of approximately 50 ml capacity and 125 ml capacity, used as evaporating dishes.

E-3.5.1.4 Glass beakers, one litre capacity and 250 ml capacity.

E-3.5.1.5 Withdrawal pipette, long stem 20 ml capacity bulb pipette, marked at 10 cm on the stem from the lower end; used as withdrawal pipette to collect fractions from 10 cm depth.

E-3.5.1.6 Desiccator

E-3.5.1.7 Sieve, 200 mm diameter to separate coarser portion from finer portion (see E-3.4.3).

E-3.5.1.8 Volumetric flask, one litre capacity.

E-3.5.1.9 Rimless glass or plastic container, 60 ml to 80 ml volume. The exact volume (that is capacity) must be known or measured accurately.

E-3.5.1.10 Glass funnel, about 15 cm long and top diameter approximately 8 cm.

E-3.5.1.11 Wash bottle

E-3.5.1.12 Bulb pipette, 25 ml capacity.

E-3.5.1.13 Stopwatch, with a resolution of 0.2 s or better.

E-3.5.1.14 Polythene circular trough, of approximately 40 cm diameter and 15 cm height.

E-3.5.1.15 Thermometer, to read temperature of the suspended mixture.

E-3.5.1.16 Enamel plate or tray, suitable to carry evaporating dishes and to keep in drying oven.

E-3.5.1.17 Tongs, for handling hot dishes.

E-3.5.1.18 Analytical balance, to weigh accurately 0.001 g or less.

E-3.5.1.19 Drying oven

E-3.5.2 Reagent (Dispersing Agent)

Dissolve 33 g of sodium hexametaphosphate and 7 g of sodium carbonate in distilled water to make 1 litre solution in a volumetric flask.

NOTE — This solution is unstable and should be prepared once in a month.

E-3.5.3 Sample for Analysis

Samples having particles in the range of silt and clay are suitable for analysis by this method. The coarser fraction should be separated by sieving and analyzed by methods described earlier for coarser particles (that is more than 0.06 mm size particles).

E-3.5.4 Preparation of Sample and Analysis**E-3.5.4.1 Wet sieving**

See **E-2** and **E-3.4.3** to ensure proper procedure of sieving and removal of organic matter, if required. *See E-3.4.3*, for wet sieving procedure to separate sands from finer portion.

Sieve approximately 100 g of properly mixed sample. Keep a polythene circular trough (*see E-3.5.1.14*) below. Continue sieving till clear water passes through the sieve. Collect all the materials and water washings passing through the sieve in the trough.

Transfer the portion retained in the sieve (that is coarser fraction) into a 250 ml beaker using water jet from a wash bottle. Wash this portion with gravity fed deionised or distilled water by decantation method. Transfer all the washed particles into a pre-weighed dry 125 ml porcelain dish. Keep the dish initially at 85 °C to 95 °C till the content of the dish appears dry. Then keep the dish in oven at 101 °C to 105 °C overnight. Next day cool the dish in a desiccator to room temperature. Weigh the dish with coarse particles. Find the mass of the coarse part present in the sample (say 'C'g). Use this part to analyse particle size distribution of the coarser fractions (*see E-2, E-3.3 and E-3.4*).

E-3.5.4.2 Removal of salt

Transfer quantitatively the portion collected in the polythene trough (after passing through the sieve) into a vertical glass jar (*see E-3.5.1.1*). Add distilled or deionised water to fill about three fourth of the jar. Stir the mixture with a glass rod to dissolve adhered salt. Allow the mixture to settle till the top portion of the water is clear. Siphon off the top clear water and discard it. Again add distilled water to fill 0.5 to 0.75 of the jar. Stir and allow to settle. Siphon off the top clear water. Repeat the procedures till particles do not settle or water does not become clear within one hour. By this procedure most of the salt is removed. The jar now contains salt free particles with about half the jar full of water. Mark the top level of the mixture; this is required to find the volume of the mixture afterwards (*see E-3.5.4.8*).

E-3.5.4.3 To find the concentration of the finer particles collected in the jar

Stir the mixture in the jar vigorously to make it homogeneous. Using a rimless plastic or glass container of known capacity say 'J' ml (*see E-3.5.1.9*), take out quickly a container full of the homogeneous mixture and pour the mixture into a 125 ml pre-weighed dry porcelain dish (*see E-3.5.1.3*). Repeat the procedure once again for duplicate measurement that is take another aliquot in a second pre-weighed porcelain dish. Again

mark the top level of the mixture in the jar (that is a second mark). Initially, dry the contents of the dishes at 85 °C to 95 °C till the contents of the dishes appear dry. Then keep the dishes in an oven at 101 °C to 105 °C overnight. Cool the dishes in a desiccator. Weigh the dishes accurately. From the difference in mass between dish plus dried sample (silt) and the empty dish compute the mass of the silt. Take the average of two sets (say 'W' g). Calculate concentration of silt (mass/volume) in the homogeneous mixture from its mass (W) and the volume of the mixture (J) taken in each porcelain dish. This gives the concentration of silt taken in the vertical jar and mass of silt taken in one container full of mixture (W/J g/ml and W grams respectively).

E-3.5.4.4 Preparation of sample in sedimentation tube

In this method, a one litre capacity 'narrow mouth measuring cylinder' (*see E-3.5.1.2*) is used as sedimentation tube. Say the capacity of the sedimentation tube = S ml (in this stated procedure S = 1 000 ml).

If the level of the mixture present in the vertical jar goes below the second marking due to evaporation loss, add distilled water to bring the level up to the second mark. Stir the mixture thoroughly to make it homogeneous. After knowing the mass of silt (W grams) present in one container (*see E-3.5.1.9*) full of mixture, take a number of containers full of mixture into a one litre capacity narrow mouth measuring cylinder (to be used as a sedimentation tube; if necessary use a funnel for pouring the mixture) so that the mass of silt taken in it will be between 5 to 10 g. Record the number of containers (say 'N') full of mixture taken and calculate the mass (in grams) of silt actually taken (say 'M' = N × W grams) into the cylinder from the vertical jar. Add 25 ml of dispersing agent (*see E-3.5.2*). Make the volume equal to one litre by adding distilled water. Insert the airtight stopper (or cork). Shake the mixture by violent agitation continuously inverting the cylinder by hand for about 2 min. Keep the mixture overnight for the dispersing agent to act. Keep evaporating dishes ready. Keep a number of 50 ml porcelain dishes (*see E-3.5.1.3*) ready equal to the number of fractions needed to be collected. These dishes shall be properly cleaned, dried, accurately weighed (to the nearest to 0.001 g) and cooled to room temperature. Keep one more evaporating dish, cleaned, dried, cooled and accurately weighed, for blank correction.

For example, if samples fractions are to be taken at three specified time intervals from zero time, say to collect 0.02 mm, 0.006 mm and 0.002 mm fractions (that is medium silt, fine silt and clay fractions respectively), keep four evaporating dishes ready; three for three fractions and one for blank correction.

Number the dishes from one to three for three fractions and the fourth one as blank.

E-3.5.4.5 Next day record the temperature of the mixture. Keeping the cylinder stoppered, shake the contents vigorously by violent agitation and continually inverting the cylinder for about 2 min. Keep the cylinder on a horizontal flat surface. Zero time is the time when agitation ceases. Record the timing. Start the stopwatch. Remove the stopper.

E-3.5.4.6 Collection of first fraction: Close the sucking end of the 20 ml bulb pipette (**E-3.5.1.5**) with forefinger. To withdraw a fraction, slowly insert the closed pipette vertically into the mixture without disturbing it till 10 cm mark of the pipette stem coincides with the surface of the mixture. Use the other hand to keep the pipette in vertical position. Following Table 4 free the suction end of the pipette just before 10 s of withdrawal time (Table 4 has been prepared from Stokes' law for particles having average relative density 2.65). For example, the withdrawal time for collection of 0.02 mm fraction at 25 °C for particles of relative density 2.65 is 247.8 s that is 4 min 8 s approximately.

Apply suction uniformly to the withdrawal pipette. Rate of withdrawal should be such that 20 ml sample is withdrawn in approximately 20 s. Care must be taken not to create any turbulence or agitation in the mixture. Fill the pipette up to its 20 ml mark. Close it using forefinger. Carefully take the pipette out and transfer the mixture quantitatively into the pre-weighed porcelain dish number 1 (of 50 ml capacity) kept ready for this purpose (*see E-3.5.4.4*).

NOTES

- 1 Sample can be removed with a pipette using suitable means.
- 2 To lower the pipette vertically into the mixture and taking it out after filling, mechanical arrangement used for fixed depth pipette can be used.
- 3 To transfer quantitatively all the particles from the pipette to porcelain dish wash the pipette with small volume of gravity fed deionized or distilled water and transfer the washings into the dish.
- 4 It is not advisable to withdraw the first fraction too early, preferably not during the first one minute. Because particles are not at rest at zero time, an error may be introduced if the first fraction is taken too early. In case it is required to collect 0.06 mm or 0.05 mm size fractions then the depth of collection should be greater so that those fractions are not required to be collected before 1 min. From Stokes' law equation (*see E-3.1.4*) the time of collection of such fractions from a greater depth to be computed.

E-3.5.4.7 Collection of next and subsequent fractions

Following the time mentioned in Table 5, collect the next and subsequent required/desired fractions following the procedure mentioned above. For example, the withdrawal time for collection of 0.006 mm and 0.002 mm fractions at 25 °C for particles of relative

density 2.65 are 45.9 min (that is 45 min 54 s) and 6.88 h (that is 6 h 52 min and 48 s) respectively.

Collect the fractions in the respective numbered dishes. Depending upon requirement or choice, fractions of different size particles are collected as per respective time intervals. The sizes of the particles collected in those fractions will be equal to and less than the given diameters.

NOTES

- 1 While analyzing multiple number of samples, the starting time that is zero time of each sample should be decided carefully so that collection time of one fraction of one sample does not coincide with the collection time of any fraction of another sample, and enough time gap is available to collect all the fractions carefully without disturbing any of the mixtures.
- 2 In one day up to 0.002 mm fractions can be collected. If time does not permit to collect all the desired or required fractions in one day, collection of finer fractions can be carried out next or subsequent days, following the collection timings as per Table 5. However, due to displacement of water by settling particles, the water tends to rise up the walls of the sedimentation tube and dissipate itself as convection currents at the top of the sedimentation column and carrying with it some of the finer particles. This leads to excess of fines at the top of the tube and an over-estimation of the fines percentage in an analysis. Due to this reason, the centrifugal method is considered to be more accurate to analyse very fine, less than 0.002 mm diameter, particles.

E-3.5.4.8 Determination of masses of individual fractions

Take the dishes, containing sample fractions, on an enamel tray or plate. Keep the plate in an oven at 105 °C over night. Next day take the plate out, transfer the dishes in a desiccator with the help of a tongs. Allow the dishes to cool to room temperature. Weigh the dishes quickly to nearest 0.001 g. From the differences in the masses of the dishes plus dried solid fractions and the masses of the respective empty dishes compute the masses of the individual fractions.

The values are corrected for dispersing reagent (blank correction). For blank correction, take 25 ml of the dispersing agent (*see E-3.5.2*) in a 1 litre volumetric flask. Add distilled water to make the volume one litre. Shake the mixture thoroughly to make it homogeneous. After about 1h take 20 ml of the diluted solution of the dispersing agent in a pre-weighed 50 ml capacity porcelain dish kept for blank correction purpose (*see E-3.5.4.4*). Dry the content of the dish in an oven for overnight at 101 °C to 105 °C. Cool the dish to room temperature in a desiccator. Weigh the dish quickly to nearest 0.001 g. Subtract mass of the empty dish from this value. This will give mass of the dispersing agent present in 20 ml of the diluted solution. Subtract this value from the masses of the individual fractions determined above. This will give corrected masses of the individual fractions. Let the corrected masses of the individual fractions are ' g_i ' gs where 'i' stands for fraction number.

Empty the vertical jar (which was used in **E-3.5.4.2**), wash with water. Fill the jar with water up to the first mark that is marking for the top level of the mixture taken in it. Measure volume of the water using a measuring cylinder (say 'V ml, which gives volume of the silt water mixture taken in the jar). From the concentration of silt taken in the vertical jar (W/J g/ml as determined in **E-3.5.4.3**) and volume of the mixture in the jar (V ml) as determined by this method, calculate total silt content in the mixture taken in the jar. This gives total mass of the finer portion of the sample (say ' $F = (V \times W)/J$ g. From the mass of the coarser portion of the sample, say C g (see **E-3.5.4.1**), calculate the total mass of the sample taken for particle size analysis purpose, say $T = (F+C)$ g.

E-3.5.5 Calculations

Calculate the cumulative percentage of the finer fractions with respect to the whole sample (that is fine plus coarse part), from the formula:

$$\frac{g_i \times S}{v_i \times M} \times \frac{F}{T} \times 100 \text{ percent}$$

where

g_i = corrected mass of individual fractions, in g (see **E-3.5.4.8**);
 S = volume of sample in sedimentation tube, in ml (see **E-3.5.4.4**; in the present procedure $S = 1000$ ml);
 v_i = volume of individual fractions in ml (see **E-3.5.4.6**; in the present procedure $v_i = 20$ ml);
 M = mass of silt taken in sedimentation tube (that is measuring cylinder), in g (see **E-3.5.4.4**);
 F = mass of the finer portion of the sample taken in vertical jar, in g (see **E-3.5.4.8**); and
 T = total mass of the sample taken for purpose of particle size analysis, in g (see **E-3.5.4.8**).

NOTE — To analyse particle size distribution of silt and clay by pipette method for samples having relative density other than 2.65 or to analyse at temperatures beyond 35 °C use Stoke's law equation (see **3.1.4**) to collect fractions, from 10 cm or some other suitable depth, at appropriate times.

Table 5 Rate of Settling of Particles at Various Temperatures (Average Relative Density as 2.65)
(Clause 3.5.4.7)

Sl. No.	Temper- ature	Diameter, mm																		
		0.001 0.002 0.003 0.004				0.005 0.006 0.007 0.008 0.009 0.01					0.02 0.03 0.04 0.05 0.06 0.07 0.08 0.09 0.10									
		in hours					in minutes					in seconds								
(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)	(9)	(10)	(11)	(12)	(13)	(14)	(15)	(16)	(17)	(18)	(19)	(20)	(21)
i)	10	40.41	10.10	4.49	2.53	97.0	67.4	49.5	37.9	29.9	24.2	363.7	16.17	90.9	58.2	40.4	29.7	22.7	18.0	14.5
ii)	11	39.30	9.83	4.37	2.40	94.3	65.5	48.1	36.8	29.1	23.6	353.7	157.2	88.4	56.6	39.3	28.9	22.1	17.5	14.1
iii)	12	38.19	9.55	4.24	2.39	91.7	63.6	46.8	35.8	28.3	22.9	343.7	152.8	85.9	55.0	38.2	28.1	21.5	17.0	13.7
iv)	13	37.17	9.29	4.13	2.32	89.2	61.9	45.5	34.8	27.5	22.3	334.5	148.7	83.6	53.5	37.2	27.3	20.9	16.5	13.4
v)	14	36.15	9.04	4.02	2.26	86.8	60.2	44.3	33.9	26.8	21.7	325.3	144.6	81.3	52.1	36.1	26.6	20.3	16.1	13.0
vi)	15	35.22	8.80	3.91	2.20	84.5	58.7	43.1	33.0	26.1	21.1	317.0	140.9	79.2	50.7	35.2	25.9	19.8	15.7	12.7
vii)	16	34.29	8.57	3.81	2.14	82.3	57.2	42.0	32.1	25.4	20.6	308.6	137.2	77.2	49.4	34.3	25.2	19.3	15.2	12.3
viii)	17	33.43	8.36	3.71	2.09	80.2	55.7	40.9	31.3	24.8	20.1	300.8	133.7	75.2	48.1	33.4	24.6	18.8	14.9	12.0
ix)	18	32.56	8.14	3.62	2.04	78.1	54.3	39.9	30.5	24.1	19.5	293.0	130.2	73.3	46.9	32.6	23.9	18.3	14.5	11.7
x)	19	31.76	7.94	3.53	1.98	76.2	52.9	38.9	29.8	23.5	19.1	285.8	127.0	71.5	45.7	31.8	23.3	17.9	14.1	11.4
xi)	20	30.98	7.75	3.44	1.94	74.4	51.6	37.9	29.0	23.0	18.6	278.8	123.9	69.7	44.6	31.0	22.8	17.4	13.8	11.2
xii)	21	30.24	7.56	3.36	1.89	72.6	50.4	37.0	28.3	22.4	18.1	272.1	121.0	68.0	43.5	30.2	22.2	17.0	13.4	10.9
xiii)	22	29.52	7.38	3.28	1.85	70.9	49.2	36.2	27.7	21.9	17.7	265.7	118.1	66.4	42.5	29.5	21.7	16.6	13.1	10.6
xiv)	23	28.83	7.21	3.20	1.80	69.2	48.1	35.3	27.0	21.4	17.3	259.5	115.3	64.9	41.5	28.8	21.2	16.2	12.8	10.4
xv)	24	28.17	7.04	3.13	1.76	67.6	47.0	34.5	26.4	20.9	16.9	253.6	112.7	63.4	40.6	28.2	20.7	15.8	12.5	10.1
xvi)	25	27.53	6.88	3.06	1.72	66.1	45.9	33.7	25.8	20.4	16.5	247.8	110.1	61.9	39.6	27.5	20.2	15.5	12.2	9.9
xvii)	26	26.92	6.73	2.99	1.68	64.6	44.9	33.0	25.2	19.9	16.2	242.3	107.7	60.6	38.8	26.9	19.8	15.1	12.0	9.7
xviii)	27	26.32	6.58	2.92	1.65	63.2	43.9	32.2	24.7	19.5	15.8	236.9	105.3	59.2	37.9	26.3	19.3	14.8	11.7	9.5
xix)	28	25.75	6.44	2.86	1.61	61.8	42.9	31.5	24.1	19.1	15.4	231.7	103.0	57.9	37.1	25.7	18.9	14.5	11.4	9.3
xx)	29	25.19	6.30	2.80	1.57	60.5	42.0	30.9	23.6	18.7	15.1	226.8	100.8	56.7	36.3	25.2	18.5	14.2	11.2	9.1
xxi)	30	24.66	6.16	2.74	1.54	59.2	41.1	30.2	23.1	18.3	14.8	221.9	98.6	55.5	35.5	24.7	18.1	13.9	11.0	8.9
xxii)	31	24.15	6.04	2.68	1.51	57.9	40.2	29.6	22.6	17.9	14.5	217.3	96.6	54.3	34.8	24.1	17.7	13.6	10.7	8.7
xxiii)	32	23.65	5.91	2.63	1.48	56.7	39.4	29.0	33.3	17.5	14.2	212.8	94.6	53.2	34.0	23.6	17.4	13.3	10.5	8.5
xxiv)	33	23.16	5.79	2.57	1.45	55.6	38.6	28.4	21.7	17.2	13.9	208.5	92.7	52.1	33.4	23.2	17.0	13.0	10.3	8.3
xxv)	34	22.70	5.67	2.52	1.42	54.5	37.8	27.8	21.3	16.8	13.6	204.3	90.8	51.1	32.7	22.7	16.7	12.8	10.4	8.2
xxvi)	35	22.24	5.56	2.47	1.39	53.4	37.1	27.2	20.9	16.5	13.3	200.2	89.0	50.1	32.0	22.2	16.3	12.5	9.9	8.0

ANNEX F

(Clause 9)

DETERMINATION OF THE RELATIVE DENSITY OF SEDIMENT

F-1 PROCEDURE

F-1.1 Dry the sample in a drying oven at a temperature of 101–105 °C until a reasonably constant mass is obtained. Allow it to cool in a desiccator and keep until required for the determination.

F-1.2 Wash a density bottle (or pycnometer) of suitable size (say 10 cm³ or 25 cm³) with gravity fed deionized or distilled water. Fill it completely with distilled water at room temperature (which should be recorded to the nearest 0.1 °C), insert the stopper and wipe off any superfluous water, dry the outside with lens cleaning paper or chamois leather. During the procedure, ensure that no air bubbles are present. Weigh it to at least the nearest 1 mg and record the mass of the density bottle (or pycnometer) filled with distilled water (m_2).

NOTE — This procedure (filling and weighing) should be performed accurately and rapidly.

F-1.3 Open the stopper. Empty the density bottle. Dry it and its stopper in an oven at 101 °C to 105 °C until a reasonably constant mass is obtained. Cool the density bottle and its stopper to room temperature. Insert the stopper and then accurately weigh the bottle to at least the nearest 1 mg. Repeat this procedure until a constant mass is obtained. Record the mass (m_1) of the empty density bottle.

F-1.4 Open the stopper. Add about 2 g to 5 g of dry

sediment in the density bottle. Insert the stopper. Wipe off all the particles, if any, from the outer surface of the density bottle. Weigh and record the mass of the density bottle partially filled with the sample (m_3).

F-1.5 Open the stopper. Fill the partially filled density bottle completely with distilled water by means of a pipette. Insert the stopper, wipe off the superfluous water, dry the outside with lens cleaning paper or chamois leather, weigh accurately and record the mass of the density bottle filled with sample plus distilled water (m_4).

F-2 EXPRESSION OF RESULTS

Calculate the relative density from the formula:

$$\frac{m_3 - m_1}{(m_3 - m_1) - (m_4 - m_2)}$$

where

- m_1 = mass of the density bottle (or pycnometer) (see F-1.3);
- m_2 = mass of the density bottle filled with distilled water (see F-1.2);
- m_3 = mass of the density bottle partially filled with the sample (see F-1.4); and
- m_4 = mass of the density bottle filled with distilled water plus sample (see F-1.5).

ANNEX G

(Clause 10)

DETERMINATION OF THE PARTICLE SIZE DISTRIBUTION CHARACTERISTICS

G-1 GENERAL

G-1.1 Obtaining summary information on particle size distribution characteristics involves estimation of certain descriptive statistics that are representative of the data for which frequency distribution tables have been prepared (see 8.3). Most important of such statistics are the mean, epitomizing the central tendency, and the standard deviation embodying the variability of particle sizes.

G-2 DETERMINATION OF PARTICLE SIZE CHARACTERISTICS

G-2.1 Arithmetic Mean Particle Diameter

A commonly used measure of the central tendency is the arithmetic mean. It is the sum of the values recorded in a series divided by the number of values. For a grouped distribution:

$$\bar{D} = \frac{\sum p_i D_i}{\sum p_i}$$

where

 \bar{D} = mean diameter; D_i = mid-value of class interval i ; and P_i = percentage of the mass of the sample in the i th class interval.

G-2.2 Geometric Mean Particle Diameter

Since the particle size distribution of bed material is most often log normal, the location parameter is the mean of $\log D_g$ and the estimation is:

$$\log D_g = \frac{\sum p_i \log D_i}{\sum p_i}$$

where

 p_i = percentage of the mass of the sample in the i th class interval; and D_i = mid-value of i th class interval of particle diameters.

G-2.3 Median

The median, usually denoted by D_{50} , is another measure of central location, widely used to represent the general nature of the particle size of samples. D_{50} is that diameter for which the aggregate mass of all particles smaller than this size is 50 percent of the total mass. Median is a convenient measure of the central tendency of particle size data for which cumulative percentage curve is available. In practice D_{50} can be obtained from the cumulative curve as the point of intersection of the horizontal at 50 percent of the percentage scale with the curve.

G-2.4 Percentile Points

Other percentile points entering into the concepts of bed load motion are D_{65} , D_{35} , D_{90} and D_{10} . These percentile points may be obtained in a similar way as the median. D_{65} and D_{35} represent the diameters of particles of which 65 percent and 35 percent respectively are finer by mass.

G-2.5 Example of Determination of Particle Size Distribution Characteristics

The procedures of computing some of the location parameters are partially set out in Table 6.

Geometric mean particle diameter:

$$D_g = \text{Antilog} (-44.503 \ 3/100) = 0.359 \text{ mm}$$

Table 6 Computation of Geometric Mean Diameter and Arithmetic Mean Diameter
(Clause G-2.5)

Sl No.	Size Range mm	Percentage of Mass of Sample p_i	D_i mm	$\log D_i$	$p_i D_i$	$p_i \log D_i$
(1)	(2)	(3)	(4)	(5)	(6)	(7)
i)	4-2	0.5	3.0	0.477 1	1.50	0.238 6
ii)	2-1	5.6	1.5	0.176 1	8.40	0.986 2
iii)	1-0.5	11.7	0.75	- 0.124 9	8.78	- 1.461 3
iv)	0.5-0.25	53.7	0.375	- 0.426 0	20.14	- 22.876 2
v)	0.25-0.125	26.4	0.187	- 0.728 2	4.94	- 19.224 5
vi)	0.125-0.062 5	2.1	0.093	- 1.031 5	0.20	- 2.166 1
					$\sum p_i D_i$ 43.96	$\sum p_i \log D_i$ - 44.5033

Mean particle diameter:

$$\bar{D} = 43.96/100 = 0.44 \text{ mm}$$

G-3 DETERMINATION OF STANDARD DEVIATION

G-3.1 The parameter most commonly used to characterize particle size distribution is the standard deviation, which is the square root of the average of squares of the deviations from the mean.

$$S_D = \sqrt{\frac{\sum (D_i - \bar{D})^2 p_i}{\sum p_i}}$$

The variance of $\log D$, namely σ^2 , may be estimated from the formula:

$$\sigma^2 = \frac{1}{(n-1)} \sum_{i=1}^n (\log D_i - \log D_g)^2$$

where σ is the standard deviation of $\log D$.

Since the particle size distribution is more often log normal, using standard normal probability curve, an approximation of the standard deviation of particle size value is obtained as:

$$S_{D_g} = \frac{1}{2} (D_{84} - D_{16})$$

where D_{84} is that diameter for which the aggregate mass of all particles smaller than this size is 84 percent of the total mass. Likewise, D_{16} is that diameter for which the aggregate mass of all particles smaller than this size is 16 percent of the total mass. They can be obtained (approximately) by interpolation from col 2 of Table 6. Alternatively, estimates of D_{84} and D_{16} can also be obtained from the particle size distribution curve plotted on log-normal paper (see for example Fig. 15, which is based on another set of data); which in turn can be used for computing standard deviation.

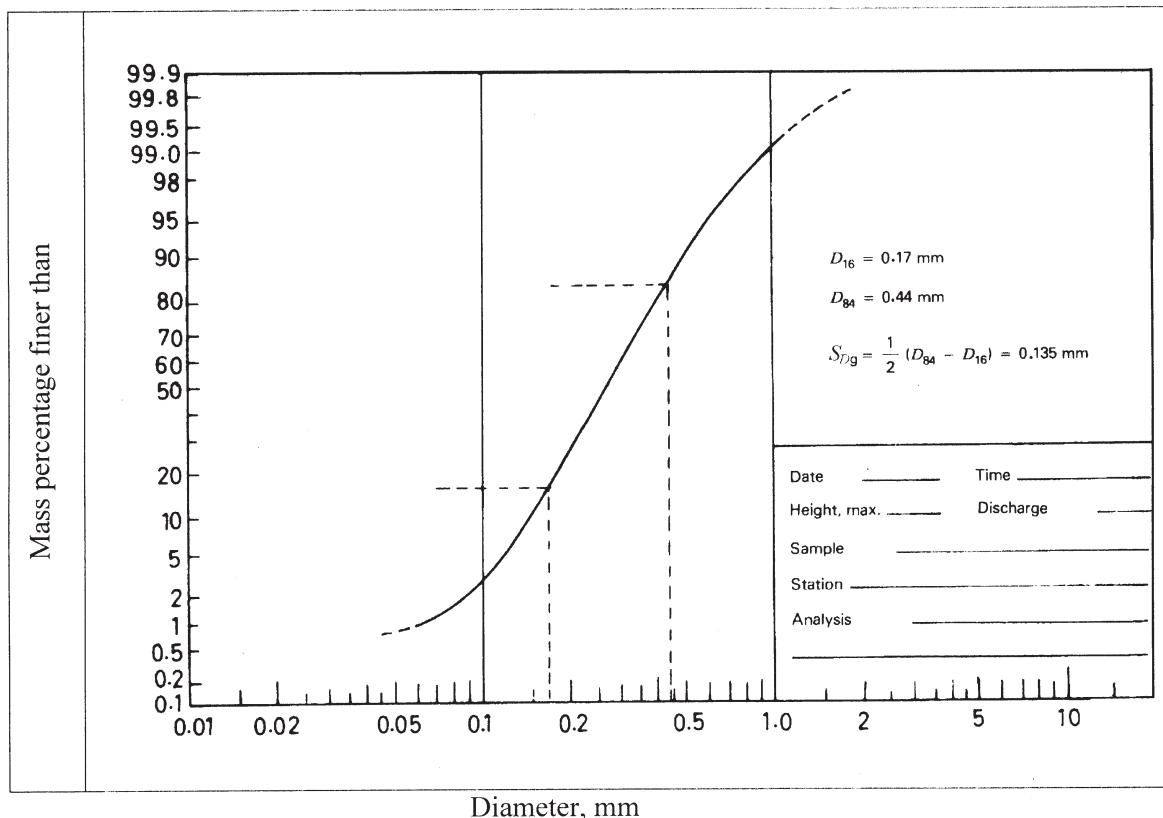


FIG. 15 DETERMINATION OF PERCENTILE POINTS AND GEOMETRIC STANDARD DEVIATION

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